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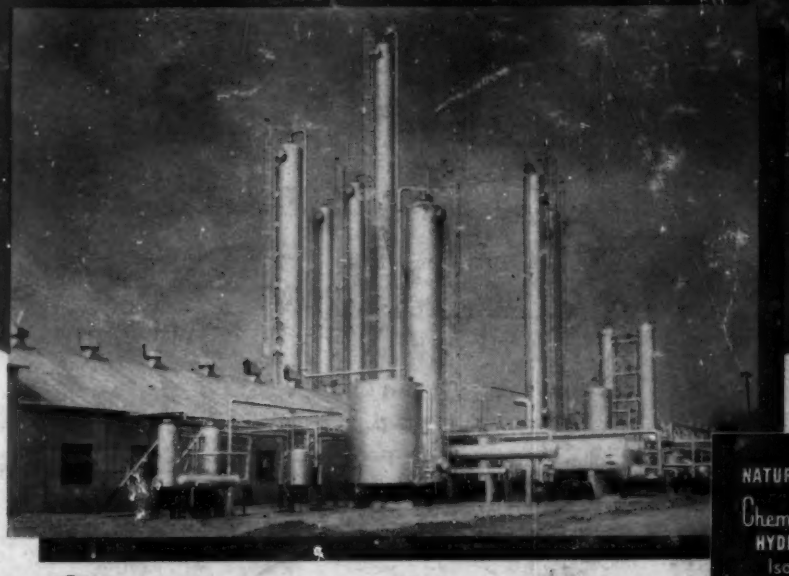

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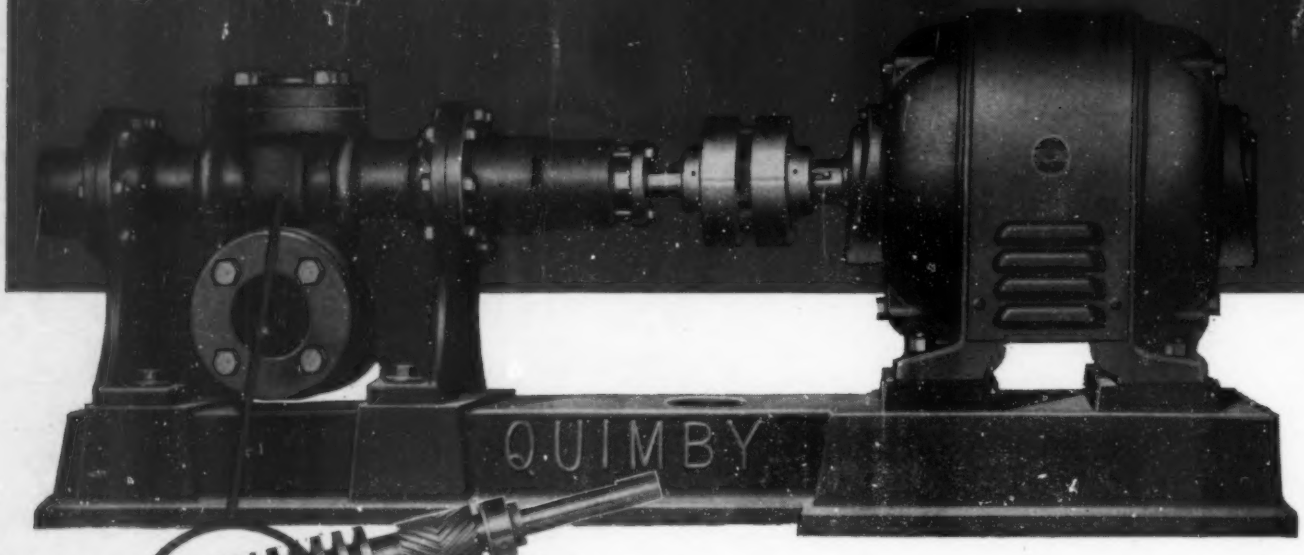

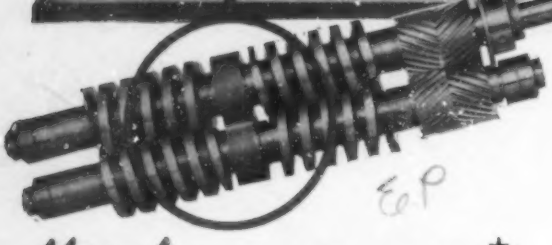

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SPECIALIZED FOR THE OIL REFINING INDUSTRY, INCLUDING PETRO-CHEMICAL ENGINEERING, PETROLEUM SYNTHETICS AND NATURAL GASOLINE

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THE LOOK BOX

IN THE matter of labor the new Congress faces the precise task of leaving men with their right to strike, while protecting the public from the strike. It is a fine distinction and it calls for statesmanship rather than vengeance.

A Matter Of Freedom

The constitutional right of each citizen of the United States includes the privilege of walking off the job as he pleases. This is fundamental and it can not be circumscribed, otherwise men will lose freedom in other matters. And freedom is not to be numbered or listed by degrees. The Four Freedoms had a run of publicity but it is better to have freedom without numeration or definition.

But in the labor movement of the United States over the past dozen years men have been giving up that bit of freedom—the right to strike. Individuals do not strike. Strikes are called for them. Men obey in fear, rather than in freedom.

What has taken place is the creation of monopoly in labor, which can be even more vicious than monopoly of capital. In a monopoly of capital, progress can be halted. An exercise of the monopoly of labor can bring hunger and privation.

Herein the laws need revision. It should not be legal for a man or a set of men to gain control in the labor market, just as it should not be legal for men to gain control of the monetary system or the food supply of a nation. Regardless of their base of operation, men become autocratic when they hold such privileges. The quicker the system is abolished, the quicker freedom will be less circumscribed.

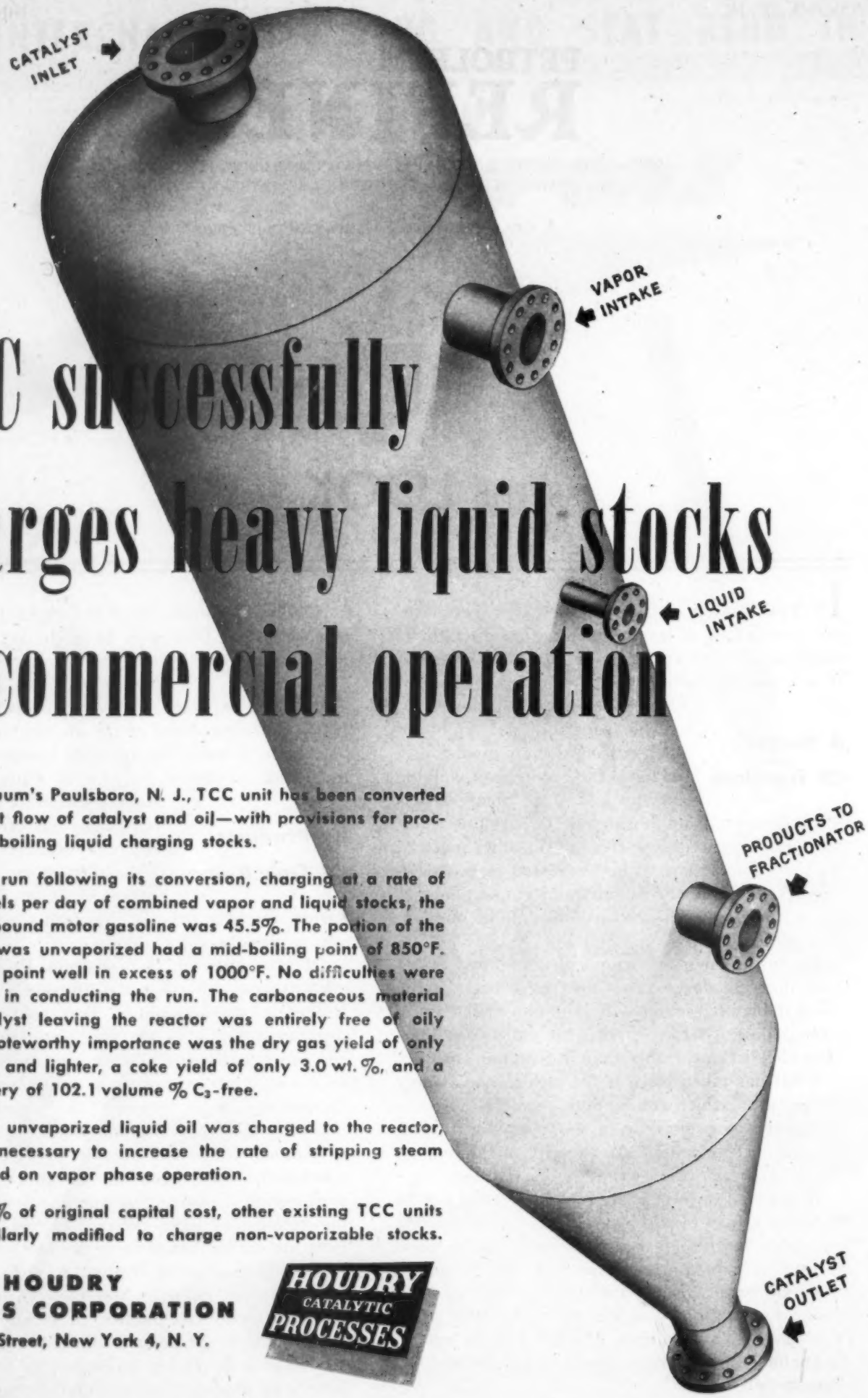
That men should have the right to strike is unquestioned. That men have the right to force men into a strike is indefensible.

AS WAS expected science is now evaluating the beneficial by-products of the atomic bomb. Before the recent chemical-engineering symposium of the American Chemical Society in Pittsburgh, Dr. Manson Benedict said the development has created a new field of organic chemistry. These peacetime materials, made chiefly of fluorine and carbon, include plastics, lubricants and solvents, which already are available.

By-Products Of Oak Ridge

Dr. Benedict of Hydrocarbon Research, Inc., who designed the gaseous diffusion plant at Oak Ridge, is of the opinion that concentration of uranium 235 may be the only separation to which the process may be advantageously applied. But he outlined advancements in equipment, which in themselves may outweigh the expenditure involved in the Oak Ridge project.

"Among the most notable developments," he continued, "have been the advances in vacuum engineering. Construction specifications and welding techniques have been developed for making intricate equipment of large volume vacuum-tight to an unprecedented degree. A new mass spectrometer leak detector and new vacuum testing methods make leak hunting in vacuum systems a rapid, simple procedure instead of a tedious, laborious task. These developments are bound to benefit all aspects of vacuum technology and may,



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Socony-Vacuum's Paulsboro, N. J., TCC unit has been converted to concurrent flow of catalyst and oil—with provisions for processing high boiling liquid charging stocks.

On the first run following its conversion, charging at a rate of 10,000 barrels per day of combined vapor and liquid stocks, the yield of 10 pound motor gasoline was 45.5%. The portion of the charge that was unvaporized had a mid-boiling point of 850°F. with an end point well in excess of 1000°F. No difficulties were encountered in conducting the run. The carbonaceous material on the catalyst leaving the reactor was entirely free of oily matter. Of noteworthy importance was the dry gas yield of only 3.9 wt. % C_3 and lighter, a coke yield of only 3.0 wt. %, and a liquid recovery of 102.1 volume % C_3-free.

Even though unvaporized liquid oil was charged to the reactor, it was not necessary to increase the rate of stripping steam formerly used on vapor phase operation.

For about 1% of original capital cost, other existing TCC units can be similarly modified to charge non-vaporizable stocks.

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for instance, lead to interesting developments in high voltage accelerators and other branches of applied physics.

"A continuous mass spectrometer for process analysis was developed for recording the concentration of contaminants with substantially no time lag. This instrument is bound to prove useful in many gas processes where it is important to have a rapid, continuous record of gas concentrations.

"The development of vacuum-tight globe valves which can be opened and closed hundreds of times before seat leakage sets in will be of great importance to the low-pressure process industries.

"The knowledge gained by pump manufacturers in designing high-ratio centrifugal compressors has notably advanced the art of designing similar machines for air, refrigerants and other gases.

"The development of methods for applying an adherent internal nickel plate to steel pipe should be of great value in many other processes treating corrosive chemicals."

What Is a Work Week?

CARRIED to the extreme now indicated the working day can be made to include the time spent in traveling between home and the shop or office. In that instance, suburban living will become profitable, as well as attractive, since the individual thus situated can draw pay for the hours spent enjoying the scenery between home and place of activity.

That some activities involve a preparation that should be given financial consideration is admissible. These, however, should be made a part of contracts. But to take one such instance and its court decision as a pretext for moving against the corporations in a phalanx is little short of cowardly. Moves like that are one of the reasons for the trend in elections, things to be avoided.

As for what to do, there is the instance of the Pittsburgh Equitable Meter Division of Rockwell Manufacturing Company, which has answered a "portal-to-portal" court action with a suit of its own. The issue has been summed up as:

"The issue basically is what constitutes a work week. We have always treated our employes and the duties they perform in good faith and with an open mind and have carefully seen to it that each employe was fully compensated. If, after all these years, it is necessary to redetermine what constitutes a forty-hour work week, then the court should have before it all the facts. It has been recognized for some time that some of our employes, in spite of a written contract, have not worked an eight-hour day or a forty-hour week but have expected compensation for eight hours each day and forty hours each week. Now that the issue has been put squarely before us it is our intention to show the extent of loss to the company arising out of slowdowns and the admitted refusal to maintain production. This is regrettable but as chief executive officer of the company, I must be as fully aware of the rights of the stockholders as I am of the right of employes. Each group must be and will be protected but neither group will be made to suffer for the benefit of the other. All that should be expected of either is a fair day's work for a fair day's pay."

As to Rubber

TOWARD the end of this year production of natural rubber is due to reach a level equal to total consumption. Then will come the critical moment for the chemical-rubber industry and its role in national affairs. It has been the attitude of PETROLEUM REFINER that a substantial plant capacity be maintained, sufficient to make sure that this nation never again face the prospect it looked upon when Singapore fell.

How to maintain that much capacity for making chemical rubber is not a question that permits simple answer. Certainly it will not be wise simply to continue operating some plants as they are now operating. Subsidy will keep plants operating; it will not keep progress operating. Chemical rubber needs the advantage of competition from the laboratory into pilot plants, into commercial plants and into sales.

Good Solid United States

BY WHAT reasoning "183 miles of large-diameter steel line pipe" came to be classed as merchandise such as UNRRA should distribute to the ill-clothed and ill-fed of the world is beyond comprehension. Such was the case, however, and is how the matter of using this pipe for the big gas line from West Texas to California got into the Federal District Court of West Texas.

It seems that the United States Treasury had placed the order for the pipe with A. O. Smith Corporation. When it was needed for the Texas-California gas line, questions were shouted. The order had been placed for UNRRA, which had planned delivery to the Ukraine district of USSR. When protests became numerous, the Treasury Department cancelled its order. Still the corporations which needed the pipe and would have been affected by its delivery to Russia went through with the litigation.

Down in a lower court the welfare of the people of the United States took higher rating than did the needs of the people of Russia, so the big gas line is going to have first call on pipe from one mill, all this activity of giving a bottle of milk to each Hindu and a pair of rayon pants to each Hottentot to the contrary notwithstanding.

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History, Source, and General Nature of

Asphaltic Bitumen

A. J. HOIBERG
Director of Asphalt Research, Lion Oil Company

THE widespread use of asphalts in modern engineering practice is shown by the large volume supplied annually. Sales of nearly 7 million short tons of petroleum asphalt, exclusive of road oil, and about 800 thousand tons of natural asphalts were reported by the Bureau of Mines for 1944.[7] Included in this demand are products which vary from high-melting brittle asphalts used in preparation of rotogravure ink, through paint bases and roofing asphalts, to relatively fluid and ductile paving materials. Asphalts find such wide and varied use in industry because of their general nature and the refiners' ability to process them readily to have the qualities desired for specific applications. Inherent properties which permit ready handling are: (1) Asphalts are soluble in ordinary hydrocarbon solvents, as petroleum naphthas, and can be "cut-back" to paints or liquid paving materials which can be applied at ordinary temperatures. (2) Asphalts become fluid upon heating to relatively moderate temperatures, and in a heated condition can be brushed or sprayed. (3) Asphalts agitated with a soap solution or clay suspension become emulsified. Water emulsions with an asphalt content of up to 65 percent can be sprayed in road building or brushed in water-proofing applications. In all of these forms, it is found that asphalts adhere tightly to materials of construction so that once in place they tend to remain permanently.

Besides these properties which make possible their use under greatly different conditions, asphalts, composed of relatively inert hydrocarbons, are highly waterproof and show extreme resistance to deterioration upon aging. As illustrative of their remarkable preserving action, there can be shown fossilized remains of prehistoric flora and fauna from the Rancho-la-Brea asphalt pits near Los Angeles.

Early Sources and Applications

The usefulness of asphalts in constructions was realized some five or six thousand years ago when the petroleum industry was founded.[1,2,5] At that time the industry was confined to the production of heavy or non-volatile petroleum products found on the surface of the earth. Blazing gas wells were of course known and worshipped by certain religious groups in Persia. But products containing substantial quantities of volatile products were excluded from general application owing to their inflammability. As distillation methods were not discovered until the second century A.D., and not applied to petroleum until the sixth century A.D., it was impossible to render inflammable petroleums suitable for general use. Thus, the early petroleum industry was essentially a bitumen industry.

Sources of natural asphalts were scattered in the Euphrates Valley in the present Iraq and in general between the River Nile in Egypt and the Indus River in India. The Dead Sea must have been a well known source of asphalt because many ancient historians refer to it. Evidences are that at early times asphalts were materials of commerce and even spoils of war. Asphalt was used not only as a mortar but for the purpose of fixing the blades of tools into their handles and as a setting for jewels and for ornamentations to works of art. The bust of Manishtusu, King of Kish (about 3600 B.C.) was carved with the conventional design showing hair and beard, and eyes of white limestone were cemented in their sockets with bitumen.

The Babylonian Era, 2500 to 538 B.C., has given examples of the extensive use of asphalt as a mortar and water-proofing agent. Building foundations often were composed of many layers of burnt brick cemented with an asphalt mixture containing sand, gravel or clay. The porous nature of the brick caused the absorption of the asphalt, thereby forming a very firm and permanent bond.

One of the outstanding feats of engineering was performed during the reign of Queen Semiramis (about 700 B.C.). A tunnel under the Euphrates, approximately 0.6 miles long was constructed from burnt bricks coated with an asphalt mortar. Here we see asphalt playing a dual role of binding materials together and water-proofing the structure at the same time.

A century later Nebuchadnezzar constructed revetments and retaining walls of burnt brick and asphalt on the Euphrates, profiting by the experience of his father, who had used sun-dried bricks which failed to hold back the waters. Nebuchadnezzar also caused the bridging of the Euphrates near Babylon. The bridge was 370 feet long with piers of burnt bricks embedded in an asphaltic mastic. The bases of these piers were further protected by a superficial coating of asphalt.

The use of asphalt in war has been recorded by the Roman naturalist Pliny the Elder (23 to 79 A.D.). He stated that defenders of the city Samosata repelled Lucullus' attack by igniting the walls with burning maltha, a liquid bituminous substance, which could be extinguished only with damp earth.

From that time until comparatively modern times there are few references to the employment of asphalt in engineering. It was not until 1838 that a sidewalk was first constructed from asphalt on the portico of the old Merchants' Exchange Building at Philadelphia. The first modern asphalt road was

constructed in 1852, in France. Pavements shortly thereafter were also laid in London.

The first asphalt roadway in the United States was laid in 1870 in Newark, New Jersey. From this time on progress was rapid, and stimulated by observations around oil fields that where crude oil had dripped from wagons and "cured" the road surface was tightly bound together. It is strange this art known to the ancients was not rediscovered until recent times.

Present Sources of Native Asphalts

Although asphalts are still produced from deposits mentioned by the ancients, in Albania and Persia, the chief sources of supply of native asphalts and bitumens of commercial importance in most recent times are as follows[1,6]: In the West Indies, Cuban and Trinidad asphalts discovered in 1535 and 1595 are still produced extensively. In Europe deposits in Italy dating from the early Roman Empire, in Switzerland and Germany from 1712 and in France from 1797 are sources of supply. In North America deposits of Grahamite, Gilsonite and Wurtzilite were discovered in the middle of the 19th century. A deposit of Bermudez asphalt was discovered in Venezuela prior to 1883. These native asphalts which are all of solid or semi-solid consistency may be separated into two classes: (a) those which are pure or nearly pure, as Bermudez Lake asphalt, and (b) those associated with mineral matter, such as Trinidad Lake asphalt and those from Iraq, as well as a considerable number of rock asphalts found both in Europe and America. Another classification is that of asphaltites, to which the very hard asphalts such as Gilsonite, Grahamite, and Glance Pitch belong.

Bermudez Lake asphalt is obtained from a pitch lake situated in the northeastern part of Venezuela. From this lake, an irregular shaped surface of about 900 acres of an average depth of about 4 feet, the asphalt is dug out by hand-labor and moved by rail to the sea for shipping. This asphalt, which is fairly hard, is miscible with petroleum residue in all proportions and usually is softened by blending with liquid petroleum residuum to the degree desired for paving and other purposes. Besides paving, it has been used in the manufacture of emulsified asphalts and for roofing and water-proofing purposes.

The pitch lake of Trinidad was visited by Columbus in 1493 and by Sir Walter Raleigh in 1595. It covers an area of about 100 acres, the center of which is 285 feet in depth. The pitch is hard enough to be dug out with picks. From the lake it is sent to the refinery to remove gas, water, vegetable, and some of the mineral matter before export. Trinidad asphalt is largely used for paving, and was used for the first time at Newark, New Jersey, in 1870. Other uses are in the manufacture of asphalt roofings, mastics, and coatings for pipes.

There are many deposits of rock asphalts, both in Europe and this country. All are associated with a considerable proportion of mineral matter. Some of the rock asphalts are suitable for paving purposes without additions. Other uses are as floor mastics and for lining reservoirs.

Of the asphaltites, Gilsonite, mined in Utah and Colorado, is the most widely employed. Many industrial uses are known, such as a base for moulded articles, wood stains, and pipe-line coatings. Gilsonite is also valuable as a hardening agent for petroleum asphalts, especially in preparation of paint bases.

Petroleum Asphalts

By far the largest proportion of asphaltic bitumen used in this country is derived from crude oil and is known as petroleum asphalt. In 1944 petroleum asphalt represented about 90 percent of total domestic asphalt sales.[7] The three general types of petroleum asphalts are differentiated by the terms straight-run, cracked, and blown.

Straight-run asphalts are distillation residues from crude oils with the volatile gases, gasoline, naphtha, kerosine and usually with all of the gasoil and the lighter lubricating oil fractions removed overhead. Some crudes do not yield asphaltic residues on distillation, but reduce to waxy oils which are suited for refining to highly viscous lubricating stocks. There are great differences in properties of residues from different crude oils, a fact which should be considered in selection of asphalts for specific applications.

The early plants for the production of straight-run asphalts were of the direct-fired batch-still type, Figure 1. The products were taken off overhead until a

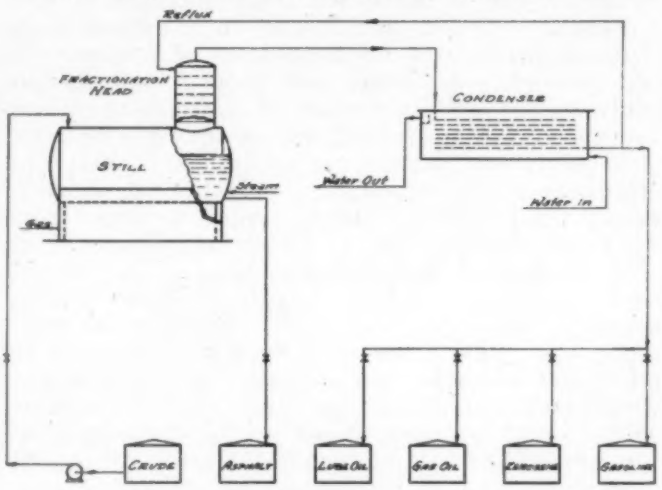

FIGURE 1
Batch still.

residue of the required hardness was obtained. Introduction of steam in large quantities was required to assist distillation. These batch stills were largely superseded by continuous shell stills, constructed similar to batch stills but in which the oil flowed from one unit to another, and with each receiving unit at a higher temperature. The last still of the battery discharged the asphaltic residue which was drawn off continuously through heat exchangers to receiving tanks.

The modern type of plant is the pipe-still unit, which has the advantages over shell stills of lower initial cost, economy and flexibility of operation, and greater throughput. This type of plant consists essentially of a furnace in which the crude oil is heated in a pipe coil and a fractionating column in which the heated oil is separated into the desired overhead fraction, side fractions, and a residue from the bottom of the column.

For the manufacture of asphalts, the charge to the still may be crude oil or a topped crude oil, that is, a residue obtained from crude oil after the lighter fractions, as gasoline, kerosine, and gasoil, have been removed by distillation in another unit. The chief difference in operating conditions between pipe stills and shell stills is that the oil passes through the heating zone in a very short time in the former.

Especially if asphalts of low penetration are to be produced a longer time is required in the shell still at high temperatures, conditions which favor cracking reactions. There are several different types of pipe-still plants used at present for production of asphaltic bitumen. These can be listed as atmospheric units, vacuum units, and combination atmospheric and vacuum units.

The atmospheric unit was the first of the continuous plants used. A simplified flow plan for one such unit is shown in Figure 2. The feed pump forces the crude oil, which has been dehydrated and desalted if necessary, through a heat exchanger to a

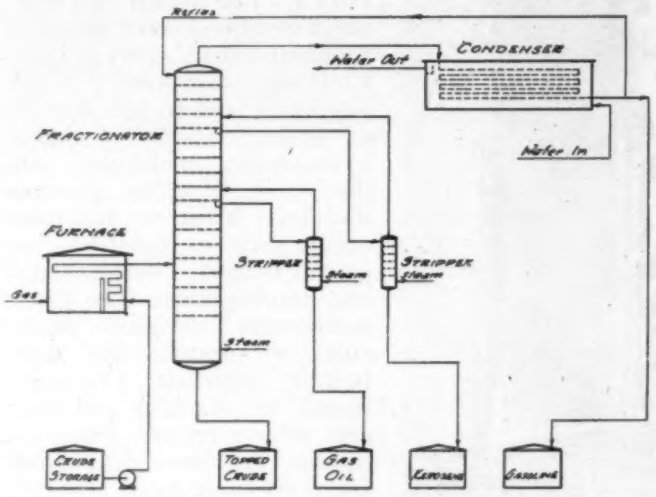

FIGURE 2
Atmospheric unit.

pipe still. The crude oil leaves the pipe heater at a temperature between 600 and 700° F., with the exact temperature dependent upon the crude and grade of residue required. Upon passing into the fractionator, a vessel about 6 feet in diameter and 30 to 40 feet in height, the pressure drops and the vapors flash off. There are three or four trays below the oil inlet and the removal of the more volatile fractions from the residue is assisted with superheated steam supplied at 500-575° F. through a spider in the bottom section of the evaporator. The residue, known as topped crude, is drawn off from the bottom of the evaporator through heat exchangers where it is cooled to about 350° F. by the crude-oil charge. From the heat exchangers, the topped crude oil flows by gravity to a receiving tank and from there can be pumped to a vacuum unit for further reduction or to storage tanks.

The vapors flashed off in the fractionator pass through from 15 to 30 trays, where suitable separation can be made of the required distillate cuts. In the case of a heavy asphaltic crude oil it is usual to take a gasoline cut of required end-point from the top of the column while two side streams yield a kerosine and gasoil or diesel oil. The end-point of the gasoline from the top of the column is controlled by refluxing part of the condensed gasoline, the rate of reflux being subjected to a temperature controller which keeps a constant temperature at the top of the column. The side streams pass to two side strippers where the more volatile fractions are removed from the gasoil and kerosine with the help of steam admitted near or at the bottom plate. The topped-crude residue from many crudes is sufficiently reduced in this type of unit to be used as a road oil and some topped crudes can by air-blowing be oxidized to roofing saturants and coatings.

Most paving grades of asphalt are refined in units equipped to operate under reduced pressure. These vacuum units are in contrast to atmospheric units which operate at normal or slightly above normal pressures and have the advantage that distillation can be carried much further without danger of crack-

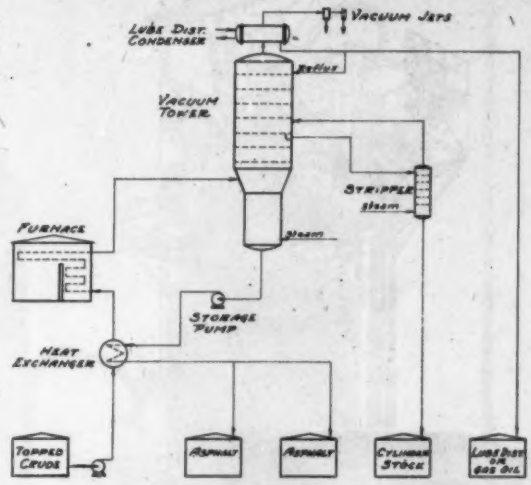

FIGURE 3
Vacuum unit.

ing, since operations are at reduced temperature. This modification is particularly suitable for the distillation of topped crude.

Figure 3 shows diagrammatically in simplified manner a usual type of a vacuum unit. Topped crude is fed to a heat exchanger and thence to a pipe still for further heating. The heated oil at the desired transfer temperature passes to a fractionator where it enters at a point about a third the way up and descends over a series of stripping trays below which superheated steam is supplied through a spider. The fractionator is under a pressure of less than 50 mm. of mercury and there is a good separation between the residual asphaltic bitumen and the distillate vapor. The asphaltic bitumen is removed from the bottom of the fractionator by means of a steam-jacketed pump through heat exchangers to storage.

The mixture of oil vapors and steam passes upwards through the fractionator section. The top of the column yields lube distillate with a cylinder-stock cut taken off as a side stream. The top of the barometric condenser is connected to a steam-jet air pump or a vacuum pump to maintain the desired vacuum on the plant. The tower is designed to allow for the larger volume occupied by the vapors under reduced pressure, and the lines must be of correspondingly larger diameter.

An example of operating conditions and yields for this type of plant, with feed of a 15° API gravity asphaltic base topped crude, is shown in the table below.

Temperatures, ° F.	
Feed inlet to pipe heater	680/690
Feed outlet	710/720
Top bubble tray of fractionating column	650/660
Pressure at top of column (mm. of mercury)	15/20
Yield of Products, percent:	
Lubricating Oil Distillate	50.0
Cylinder stock	4.5
Asphalt, 100 penetration	45.0
Loss	0.5
	100.0

Combinations of atmospheric and vacuum units

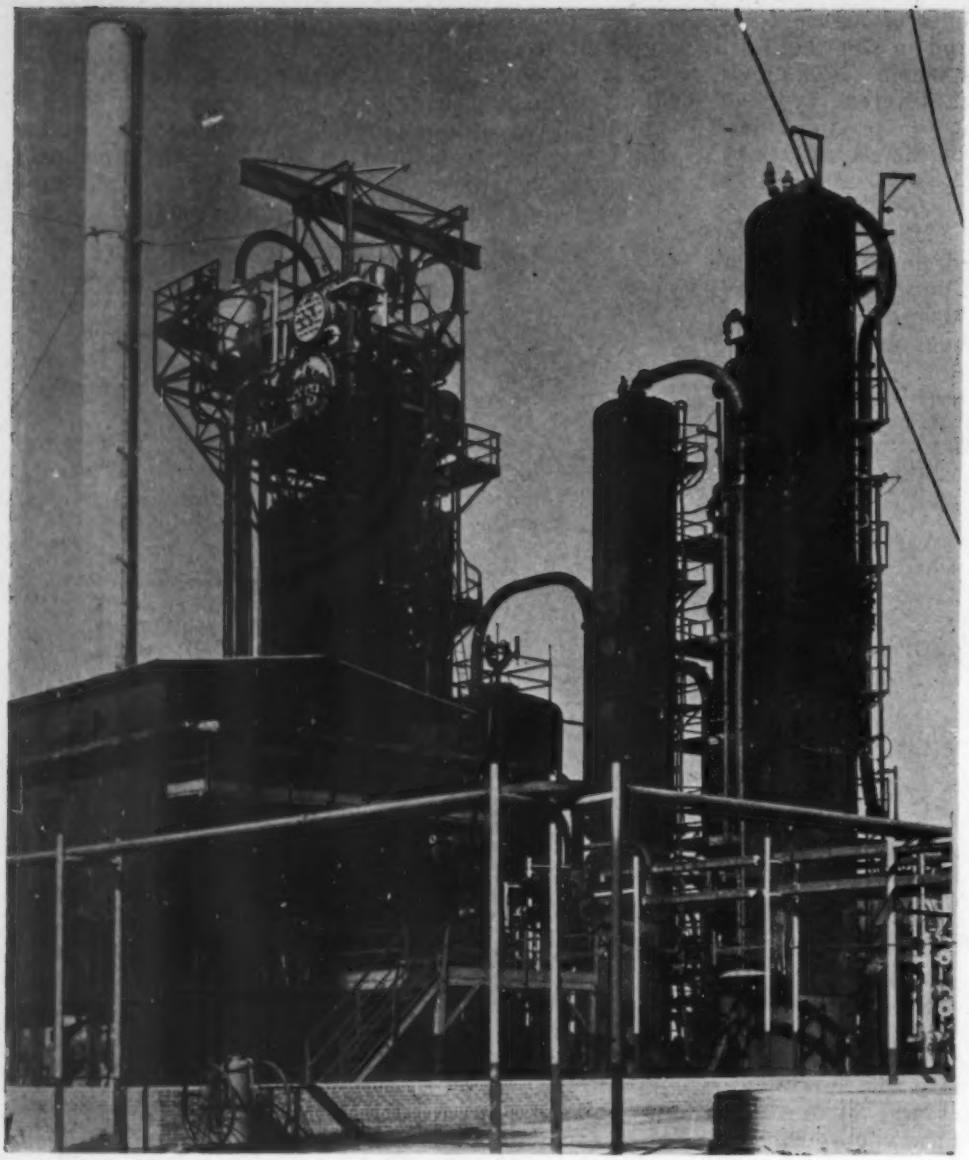

FIGURE 4
Atmospheric and vacuum unit.

are commonly employed. Figure 4 is a photograph of one type of two-stage atmospheric and vacuum unit operating on an asphaltic crude oil. The crude oil, heated in a pipe still, is flashed in an evaporator to obtain separation into two fractions, an overhead of gasoil, kerosine, and naphtha, with the residue a mixture of lubricating oil and asphalt. This residue is heated to a higher temperature by a second pass through the pipe still and is then fed into a vacuum tower. Herein separation occurs to an overhead of lubricating-oil distillate and an asphaltic residue. The overhead vapor mixture from the evaporator passes to two fractionating towers to be separated into gasoil, kerosine, and naphtha.

Asphaltic bitumen can be removed from residual oils by the addition of low-boiling hydrocarbons which cause precipitation of the asphaltic material, and retain lubricating-oil stocks in solution. The properties of asphalts separated in this way depend on the nature of the residual oil treated, the type of solvent used, and the conditions under which the treatment is carried out. With a given residual oil and at a given dilution ratio and temperature, ethane will yield a relatively high content of very soft asphaltic material, butane a lower yield of asphaltic bitumen of higher melting point, and propane, the usual solvent, will be intermediate in action. By this procedure straight-run asphalts of high softening point and low penetration can be produced which are valuable for blending operations and for special industrial applications.

Petroleum fractions become "cracked" if maintained at a temperature above about 750° F. The higher the temperature the shorter the time required for conversion. Cracking conditions are employed to lead to break-down of higher molecular weight hydrocarbon molecules into the more volatile gasoline fraction. Various reactions occur such as splitting, dehydrogenation, dealkylation, and condensation. The latter mechanism combines molecules to produce the high-boiling asphaltic products found in cracking-coil tars and which remain after the lighter gasoline and gasoil fractions have been removed in flash chambers. The cracked residua, commonly marketed as fuel and quite often as road oils, can be reduced to penetration asphalts by removal of additional distillates. Cracked residual asphalts differentiated from straight-reduced products by the Oliensis spot test also are higher in specific gravity and show a greater susceptibility to temperature change. Because of the wide variation in charging stocks and in conditions of cracking, the physical properties and service behaviors of cracked residues vary greatly. The production of cracking-coil tars has been large as by-product of straight thermal cracking, but with adoption of the newer catalytic methods the highly polymerized products formed are burned in regeneration of the catalyst and so cannot be recovered as are cracking-coil asphalts.

For many products such as roofing materials and pipe coating, in addition to the requirement of excellent resistance to weathering, the properties of high softening point and low flow at high atmospheric temperatures need to be combined with flexibility at low temperatures. In such cases a blown asphaltic bitumen is used; that is, the product obtained by oxidizing fluxes or asphalts by blowing with air under suitable conditions. In 1893, Francis X. Byerley[13] applied for a patent for the treatment of residual oil by heating the oil to a temperature of about 600° F. and by pulling air through by means of a suction pump. This appears to be the first practical attempt in the United States to make air-blown asphalt. Because of the fact that Byerley used a light Ohio residual oil and maintained this oil at

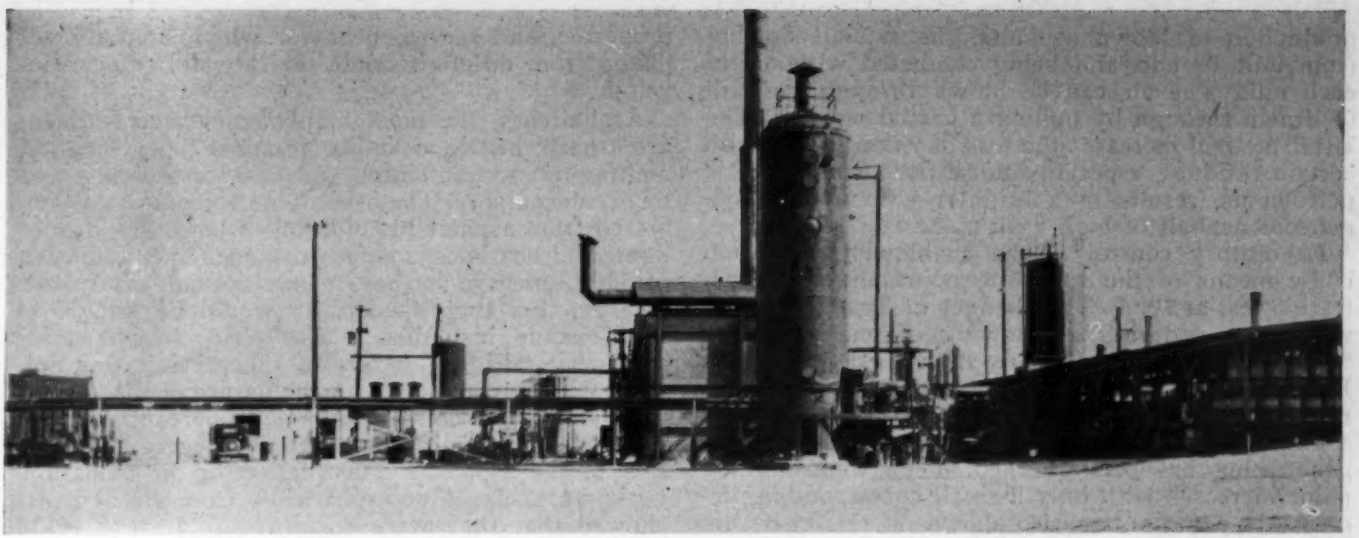

FIGURE 5
Vertical blowing still with pipe-still heater in background.

comparatively high temperatures and blew with a small quantity of air, the process was essentially distillation with the air used principally for agitation and to accelerate the distillation. There was, however, no question that the air had a chemical effect on the residuum and changed its composition.

In 1897, G. F. and G. C. K. Culmer[15] and J. W. Hayward[14] received patents for processes by which a residuum oil could be chemically changed from a liquid into a semi-solid by forcing a copious amount of air through the heated mass of oil at comparatively low temperatures. In the first practical applications of these patents, comparatively small stills holding between 1000 and 3000 gallons of residuum oil were used and the air was introduced into a heated mass of oil by a single perforated pipe laid along the bottom of the still. Because of the quality, character, and light gravity of the residuum in early blowings and because of the poor distribution of the air, the time required was from 20 to 60 hours. The early air-blown asphalts proved unsatisfactory for paving but were used successfully for industrial purposes after blending with natural asphalts, such as Gilsonite, to impart increased stability, better adhesion properties and greater ductility.

It was not until 1908 or until after the discovery of heavier crude oils of an asphaltic base in Illinois and Kansas, that the industry of producing air-blown asphalts became an important factor. These heavy semi-asphaltic oils produced residua which for the first time produced air-blown asphalts of good quality without the additional blending of natural asphalts. Shortly afterward, air-blown products were produced from the more highly asphaltic oils of Texas and California. Air-blown asphalts, with the improved processing of today, can be manufactured successfully from most residues. The present practice is to reduce the crude oil by means of pipe or tube stills and fractionating towers to a pre-determined consistency dependent on the softening point and penetration relation required in the blown product. The residuum is charged to a cylindrical still having a capacity of 6000 to 20,000 gallons. Generally from 20 to 50 cubic feet of air per minute are introduced per ton of residua. Figure 5 is a photograph of a vertical blowing still with a capacity of 12,000 gallons. The low-pressure, high-volume blowers now in use force air through the blowing coil to produce excellent distribution of air through the charge. This together with the high flash point of the residua produced by modern pipe stills has materially reduced the period of blowing. Asphalts which formerly required from 30 to 40 hours are now oxidized in from 5 to 7 hours. The temperatures at which blowing can be carried out have increased from under 425° F. to between 475° F. and 550° F.

Due to the complex structure of hydrocarbons, it has been impossible to determine definitely just what reactions take place during the conversion of the oil from a liquid to a semi-solid or a solid. It is generally agreed that oxidizing reactions occur to eliminate water and carbon dioxide and other volatile oxidation products to produce polymerized higher-melting hydrocarbons. The oxygen analyses of products produced show there is no appreciable fixation of oxygen.[10,12] The blowing reactions generate heat and for this reason once oxidation is well started no external heat is needed, in fact cooling

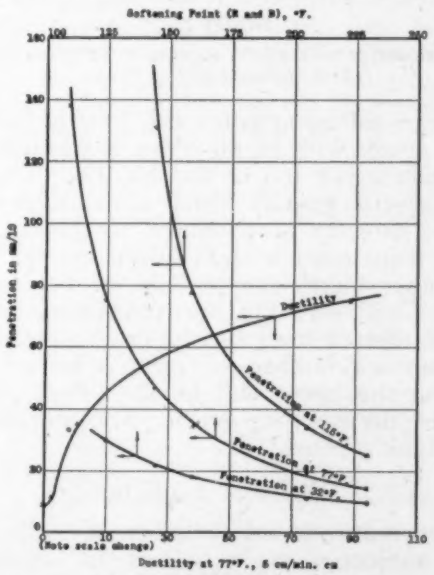

FIGURE 6
Blowing curves for a Mid-Continent roofing flux.

by circulating through a pipe still with the fires out and doors open is practiced sometimes to control the temperature.

Stills have been devised to permit the continuous

production of blown asphalts, the asphalt flowing from unit to unit and being contacted with air in each unit. The air can be blown through the still or drawn through by pulling a partial vacuum. The latter method increases the loss of volatiles and with certain residues, especially those from non-asphaltic petroleums, results in a brighter and more homogeneous asphalt of high flash point.

Laboratory control of the air-blowing operation is by means of the softening point and the penetration test at 77° F. The changes in these properties which occur during air-blowing of one particular Mid-Continent residuum are shown graphically by Figure 6. The original flux of around 500 penetration is seen to decrease rapidly in hardness as the softening point increases until a comparatively low penetration has been reached. Then the softening point increases with only a small corresponding decrease in penetration. Also shown on this chart are penetration at 32° F. and 115° F., and variation of the ductility of the asphalt at 77° F. with changes in the penetration. As the asphalt is air-blown the ductility decreases slowly and then falls rapidly as differences in structure become more pronounced. Figure 7 shows the relationship for this same blown

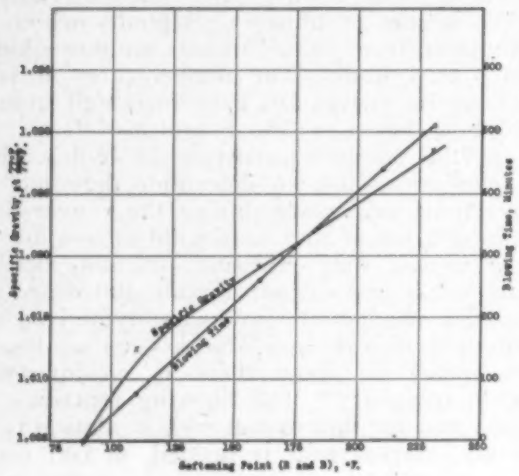

FIGURE 7

Blowing time and specific gravity changes vs. softening point for a Mid-Continent roofing flux.

flux between softening point and time of blowing in a vertical tower with an air input of 22 cubic feet of air per minute per ton of charge. The increase obtained in specific gravity which is also shown is seen to become directly proportional to the increase in softening point after a rapid initial increase.

With other fluxes the position of the curves on the graphs will shift but the trends remain similar. For blown residua from a given crude oil the general rule is that the further the base is reduced before air-blowing the lower will be the penetrations and the higher the specific gravity and ductility at a given softening point.

Nature of Asphalts

The hydrocarbons which compose asphalts have been the subject of many studies. In attempting to relate the composition of asphalts to their behavior, three major fractions commonly have been considered: asphaltenes, resins, and oils. Even though each fraction has general characteristics which are distinctive, its properties and yields vary dependent on the source and the method of analysis. For this reason correlations of fractional composition with physical properties and service behavior which hold for asphalts from different crude oils are difficult to develop.

Asphaltenes, the most highly condensed fraction, are a hard, brittle, coke-like fraction. They have no melting point but sinter and fuse on heating and finally decompose. The asphaltene component is separated from asphalt by addition of large volumes of light hydrocarbons, as for instance, upon addition of 50 volumes of hexane to one volume of asphalt. The fraction then specifically would be known as the "hexane insolubles." The yield, which varies somewhat with the nature of the precipitating solvent, will vary greatly with the nature of the asphalt and can be anywhere from 3 to 60 percent, depending upon the crude source and the degree of reduction of the asphalt by distillation or air oxidation. Ultimate analyses of asphaltenes from six asphalts showed that they were composed of from 6 to 10 percent of hydrogen, 0.6 to 8 percent of sulfur, and from 80 to 90 percent of carbon; the remainder of the molecule was ash and oxygen, with possibly nitrogen and traces of chlorine present.[10,12] Reactions with acids are closely related to those of coal as indeed are also their limited solubilities, except in highly aromatic solvents. Their mean molecular weight is known to be high but reported values vary greatly. Figures range[8] from lower than 2000 to 140,000. The asphaltene fraction itself is not homogeneous and fractions known as carbenes and carboids which show a higher carbon-to-hydrogen ratio can be separated. They are supposedly produced by condensation of highly dehydrogenated fractions as the conditions of refining become drastic, such as conversion of petroleum by cracking.

If the asphalt portion which remains after precipitation of the asphaltenes is contacted with an activated adsorber such as fullers' earth or silica gel the polar resin fraction will become adsorbed; then the naphtha extract from the adsorbent contains only the oil fraction. The resins can be recovered from the active clay with chloroform or carbon disulfide or other recommended solvents. This method gives highly variable results dependent on the degree of activity of the clay and other conditions of analysis. Methods which give more uniform results depend on selective solvents. Thus after removal of the asphaltenes a resin fraction can be precipitated by propane and other light hydrocarbons or by isobutyl alcohol or by various solvent mixtures. In all methods an arbitrary procedure must be followed exactly to obtain reproducible results. In appearance the resin fraction is usually dark brown to black in color. Analysis shows a higher ratio of hydrogen to carbon than in the asphaltene fraction. It is, however, still highly aromatic and shows a large change in viscosity with temperature. At ordinary temperatures it can be a sticky highly-ductile material and upon cooling to 40° F. harden to a brittle solid which has no ductility.

The oil component of asphalts, which usually contains some wax, has the appearance of a very heavy lubricating oil such as cylinder stock. As would be expected, the viscosity of this oil fraction from straight-reduced asphalts derived from a given crude oil is found to increase as the hardness of the asphalt increases. That is, as the distillation is carried further the less viscous and more volatile oils are removed to leave a heavier oil fraction and harder asphalt.

If desired in asphalt studies, the wax fraction can

be separated from the oils by solvent precipitation at low temperatures. The yields of the wax fraction are found to vary greatly with crude source. In small amounts wax has little effect on the behavior of asphalts. In large amounts it can be quite detrimental and cause actual non-homogeneity as shown by partial separation and aggregation of the asphaltene fraction.

As illustrative of the changes which occur during common refining processes the proportions of fractions present[3] as an asphalt is hardened, first by distillation and then by air-blowing, is shown graphically by Figures 8 and 9.

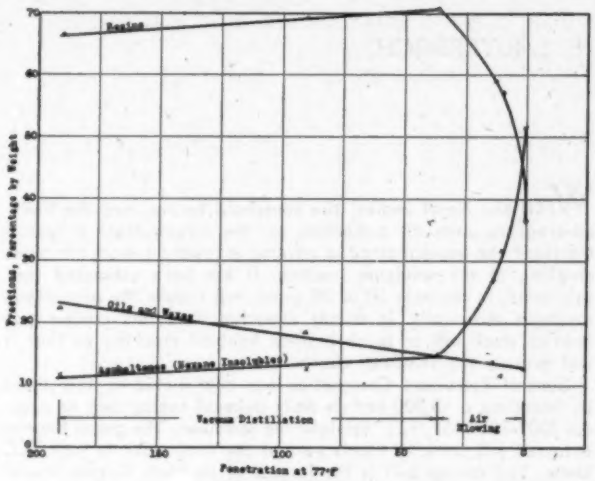

FIGURE 8

Change in fractions and penetration during distillation and air-blowing of asphalts.

Figure 8 relates the variation in fractions, expressed in percentage by weight, with the penetration at 77° F. Hardening was produced first by vacuum distillation and then by air-blowing. The oil and wax component showed the greatest fractional changes during distillation. The asphaltene and resin fractions increased slightly, but maintained a very nearly constant ratio, as would be expected with removal of the more volatile oil fractions. These changes occurred almost linearly until the air-blowing operation was started. Then the asphaltene fraction increased rapidly at the expense of the resin fraction. Only a slight loss of the oil fraction occurred, due partly to condensation to resins and partly to loss by evaporation. It can be seen that the major change in penetration is produced by distillation removal of a relatively small content of the oils.

The relation of the fractional components to the softening point of these same asphalts is shown by Figure 9. Vacuum distillation changed the softening point only slightly. Air-blowing at temperatures in the range of 500° F. increased the asphaltene content markedly and at the same time reduced the resin content to cause a relatively great increase in softening point. Thus, by means of these two operations, vacuum distillation and air blowing, it is possible to obtain within wide limits asphalts of the desired penetration and softening point for a particular application.

Examination of asphalts under the ultra-microscope as well as study of their flow properties has indicated them to be colloidal in nature; the asphaltenes instead of being in true solution exist as very small particles peptized by the resin fractions. The higher the content and the more aromatic the resin fraction, the greater the degree of dispersion of the asphaltenes. Higher temperature also increases the degree of dispersion. Swanson[11] has shown photographs of films of resins and asphaltenes deposited from benzine solutions. As the concentration of resins increased the film became clear. Katz and Beu[4] examined a series of asphalt films under the electron microscope. They concluded from micrographs at 156,000 diameters that the asphaltene particles if present are less than 65 A in diameter and suggested that the asphaltenes in most asphalts could be considered as potential colloids which easily convert into true colloids under proper conditions. An interesting and useful description of the colloid structure has been presented by Pfeiffer and Saal.[9] They consider the asphaltenes to have a marked tendency to absorb resinous hydrocarbons, with the resins of higher molecular weight held especially strongly by the asphaltenes. These resins become dissolved in resins of lower molecular weight, which in turn become surrounded by lighter constituents of less aromatic nature and so on until a gradual and nearly continuous transition to the oil and wax phase is

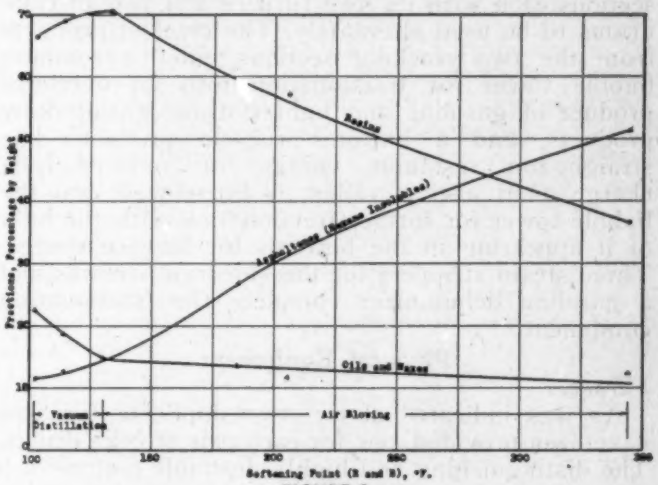

FIGURE 9

Change in fractions and penetration during distillation and air-blowing of asphalt.

reached. Thus, both the relative proportions of these three different phases and also their nature can change the properties of asphalts greatly. If the asphalt in a heated condition has been air-blown to decrease the content of resins the structure tends to become gel-like and the asphalt will show the characteristics of complex flow. Highly blown asphalts that are somewhat elastic and which if deformed slightly regain their original shape upon removal of the stress are not unusual.

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Delayed Coking Unit

At General Petroleum Corporation's Torrance Refinery

RANDAL MAASS and R. E. LAUTERBACH

WITH the diesel engine, the household burner, and the light-oil-cracking units all competing for the intermediate or gasoil fractions, the general trend in refining is toward a more extensive cracking of the residuum fraction. It has been estimated that this trend, in the next 10 or 20 years, will reduce the percentage residuum materially. It is not expected that this cracking of heavier stock will so much replace light-oil cracking as that it will provide the requisite charge stock.

General Petroleum Corporation has contributed to this trend by installing a 15,000-barrels-daily delayed coking unit to process 300"-700" SSF/122° straight-run residuum. The gasoil thereby obtained will serve to supply part of the feed stock to four TCC Units. The coking unit is the largest of its kind. Coking operations were started July 26, 1946, culminating a successful and relatively simple startup operation.

It is the intent of this paper to describe the coking process with particular emphasis on the equipment used therein. Some of the initial operating data too has been added.

This paper was read before the monthly meeting of California Natural Gasoline Association, January 2, 1947. Incidentally, through oversight, proper credit was not given to this association for "Fat Oil Deethenization," by A. M. Whistler, which appeared in Petroleum Refiner, December, 1946.

THE description of the general process may best be followed by referring to the flow sheet. The cracking portion of the coking unit is divided into two sections each with its own furnace and pair of coke drums to be used alternately. The cracked products from the two cracking sections enter a common bubble tower for fractionation into an overhead product of gasoline and lighter, three gasoil draw products, and a bottoms recycle product. The straight-run residuum charge, or reduced-crude charge as it also is called, is introduced into the bubble tower for further fractionation with the bulk of it appearing in the bottoms for furnace charge. Three steam strippers for the sidedraw streams and a gasoline debutanizer complete the fractionation equipment.

Physical Equipment

Furnaces

As was indicated above, two duplicate furnaces have been provided, one for each pair of coke drums. The distinguishing and highly desirable feature of a delayed-coking unit is the delaying of the coking until the charge reaches the soaking chamber (coke drum). To accomplish this the furnaces are designed for a low residence time and consequently a high rate of heat pickup. Because of the high heat level (600° F.) of the furnace charge, the oil tubes were restricted to the radiant section and the hotter portion of the convection section. Heat economy is realized through the use of water tubes for steam production in the colder portion of the convection bank.

In general each furnace has four separate coils which join outside the firebox into a common transfer line. The 3¼-inch outside diameter tubes are 42 feet long and have a wall thickness of 7/16-inch. Alloy tubes of 9 chrome —1.5 moly are used in the radiant section and to shield the convection bank. The remaining tubes in the convection bank are 5 chrome —0.5 moly. Heat is supplied by 16 combination oil and gas burners located in the end of the firebox.

Waste-Heat Boiler

The temperature of the furnace charge was too high to realize a good heat economy with oil tubes alone. Therefore, a waste-heat boiler, with its lower receiving temperature, has been added in the convection bank of each furnace to reduce the temperature of the flue gases. This heat pickup serves to supply some 75 percent of the plant's steam requirement. The outlet of the coils from the two furnaces enter a common steam drum from which a single pump takes suction to circulate the water through both coils. A level control maintains the water level in the drum by admitting treated water from the refinery main boiler plant feed pumps.

Coke Drums

A continuity of operation is maintained through the use of a pair of coke drums for each furnace, one drum being in coking service while the other is being decoked. Each drum is 17 feet in diameter and 80 feet in height with 61 feet of straight section. For protection against corrosion the vessels are lined with a 7/64-inch thickness of Type 405 stainless steel, the lining being resistance welded to the shell. In addition the upper half of the shell is ⅞-inch thick and the bottom half 1-inch thick. To subject a vessel of this size to the changing temperatures of blocked operations requires, of course, that it be fully stress-relieved and its welds X-rayed.

Alloy steels were used also in the principal piping to and from the coke drums. The transfer lines were fabricated from 9 chrome-1 moly pipe and the vapor lines from the drums to the bubble tower from 5 chrome-0.5 moly. Portions of the drum condensate lines, the side vapor lines, and the by-pass lines are also of alloy steel.

Bubble Tower

The 100 feet high bubble tower is 11 feet in diameter in its upper section and in its bottom section is

enlarged to 12½ feet to accommodate the increased downflow occasioned by the introduction of the intermediate reflux and the reduced crude feed. Fifteen cast-iron bubble trays are used in the upper section and in the lower section 10 Type 405 stainless steel bubble trays with 8 cast-iron baffle trays below them in the potential coking region. The shell thickness varies from 9/16- to 15/16-inch and is protected throughout by a 7/64-inch Type 405 stainless-steel strip-welded lining.

To reduce construction costs the three sidedraw steam strippers are contained in one 4½ by 56-foot shell appropriately divided into three 6-bubble-tray sections. The available hydrostatic heads dictate that the strippers be used in the same vertical order as the draw; that is, with the top draw in the top stripper, etc. The strippers are unlined and the trays are all cast iron.

It will be noted that the entire hot-oil section of the plant is protected from corrosion through the use of alloy steels. In the bubble-tower region, however, some exceptions were made after considering the corrosion tendency and ease of replacement.

Debutanizer

The design of the 4-foot, 36-bubble-tray debutanizer follows the conventional pattern. The tower is unlined and all trays are cast iron. To accommodate possible depropanization, the design working pressure was set at 330 psi.

Heat-Exchange Equipment

Some characteristics of the shell-and-tube heat exchangers are tabulated below.

Heat Exchangers

Shell Side	Tube Side	No. of Bundles	Total Surface Sq. Ft.	Tube Material
Reduced crude	Light Gasoil	2	2,480	Steel
Reduced crude	Heavy Gasoil	2	2,480	Steel
Reduced crude	Intermed. reflux	4	12,880	Steel
Reduced crude	Fuel oil	2	4,350	Steel
Stabilized gasoline	Stabilizer feed	2	1,090	Steel
Stabilizer bottoms (Stabilizer reboiler)	Intermed. reflux	1	2,000	Steel

Following our general practice open section condensing and cooling equipment is used throughout. Open sections, located in a 45-bay atmospheric cooling tower, were provided as follows:

Condensers and Coolers

Service	No. of Bundles	Total Surface Sq. Ft.	Tube Material
Bubble tower overhead	18	10,200	Admiralty brass
Light Gasoil	4	2,020	Admiralty brass
Heavy Gasoil	6	3,030	Red-brass-clad steel
Intermediate reflux	2	1,140	Admiralty brass
Stabilizer overhead	4	2,280	Admiralty brass
Stabilizer bottoms	1	530	Admiralty brass
Fuel oil	8	4,660	Red-brass-clad steel
Gland oil	2	1,060	Admiralty brass
Gland water	2	1,140	Admiralty brass

Decoking Equipment

Coke is removed from the drums by a water jet which cuts the body of coke into pieces small enough to fall into a crusher car. The crusher car breaks the pieces of coke down to a maximum dimension of 5 inches and feeds the sized coke into a sluice. The coke is sluiced with water into a suction pit from which a coke-water slurry is pumped through a 12-inch line to the storage area. In this area the bulk of the coke is separated from the water and is moved to a convenient location for shipping. The water passes through settling basins to allow any coke fines to drop out and is then returned to the unit for reuse.

The decoking water jet is supplied by a centrifugal five-stage volute pump with external interstage passages. An hydraulically balanced rotor was obtained by providing double inlets at the first stage and by placing the remaining four single-inlet stages back to back. The double inlet at the first stage also provides a low inlet velocity which permits operation at a low submergence. All impellers are of the enclosed type. A pressure breakdown bushing is provided at the inboard stuffing box so that both stuffing boxes are under suction pressure. The 4000 rpm, 1450 horsepower turbine drive is supplied with 165-pound, 100° F. superheated steam and exhausts to a 26-inch Hg. vacuum provided by a barometric condenser. Two complete pumping units are provided; one will supply 1100 gallons per minute at 1500 pounds; two in parallel will supply 1600 gallons per minute at the same pressure. Electrically-powered block valves, located in the discharge line, operate so as to prevent a shut-off against the pump. When the main block valve begins to close a smaller by-pass valve begins to open so as to discharge the water back to the suction tank. In reverse the valve operation affords the same protection. From the pump discharge the jet water flows to a manifold at the top of the coke drum.

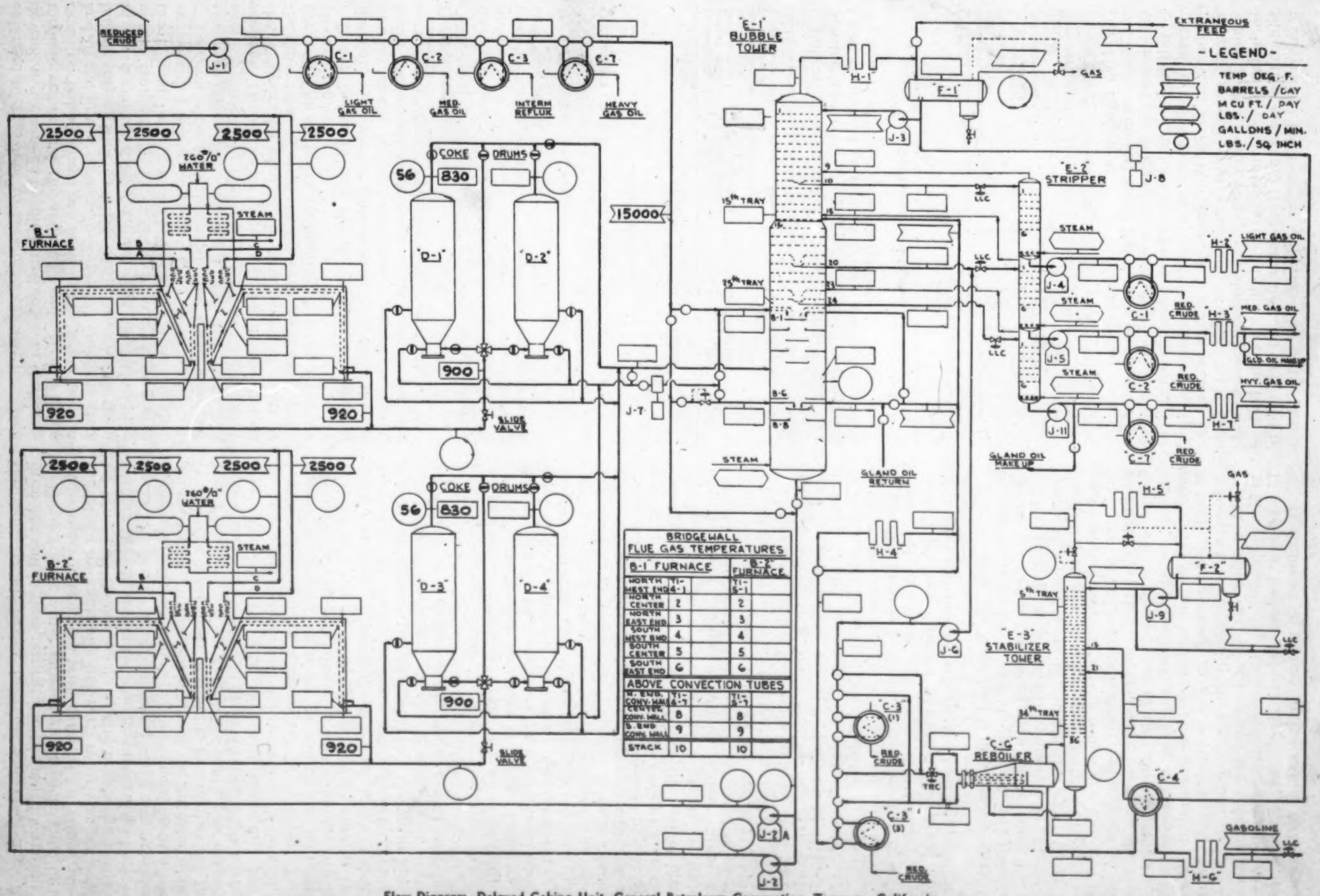
A 100-foot derrick structure is mounted over each pair of coke drums to support the drilling equipment. Each drilling assembly includes a continuous length of drill stem which is long enough to reach the whole length of the drum and to which the boring and cutting tools can be attached. The drill stem is coupled at the top to a swivel head which is rotated by an air motor and which can be raised or lowered by a steam winch. An assembly of swivel joints forms a flexible connection to supply jet water to the swivel head. Block valves have been provided in the jet-water lines to each drilling assembly. These valves are closed whenever the drill stem is removed from the drum so that no one would be injured if the high-pressure water were inadvertently turned on.

The cutting devices for removing coke are a boring tool, a reaming tool, and a cutting tool. The boring head is essentially a round ball about 10 inches in diameter which is tapped for water nozzles. One ½-inch nozzle is located in the bottom to direct a jet straight downward, and four ⅜-inch jets are symmetrically arranged about it to direct the water downward at about 45° from the vertical. Two ⅛-inch jets are directed upward to keep any coke fines from settling on top of the tool and stopping its rotation.

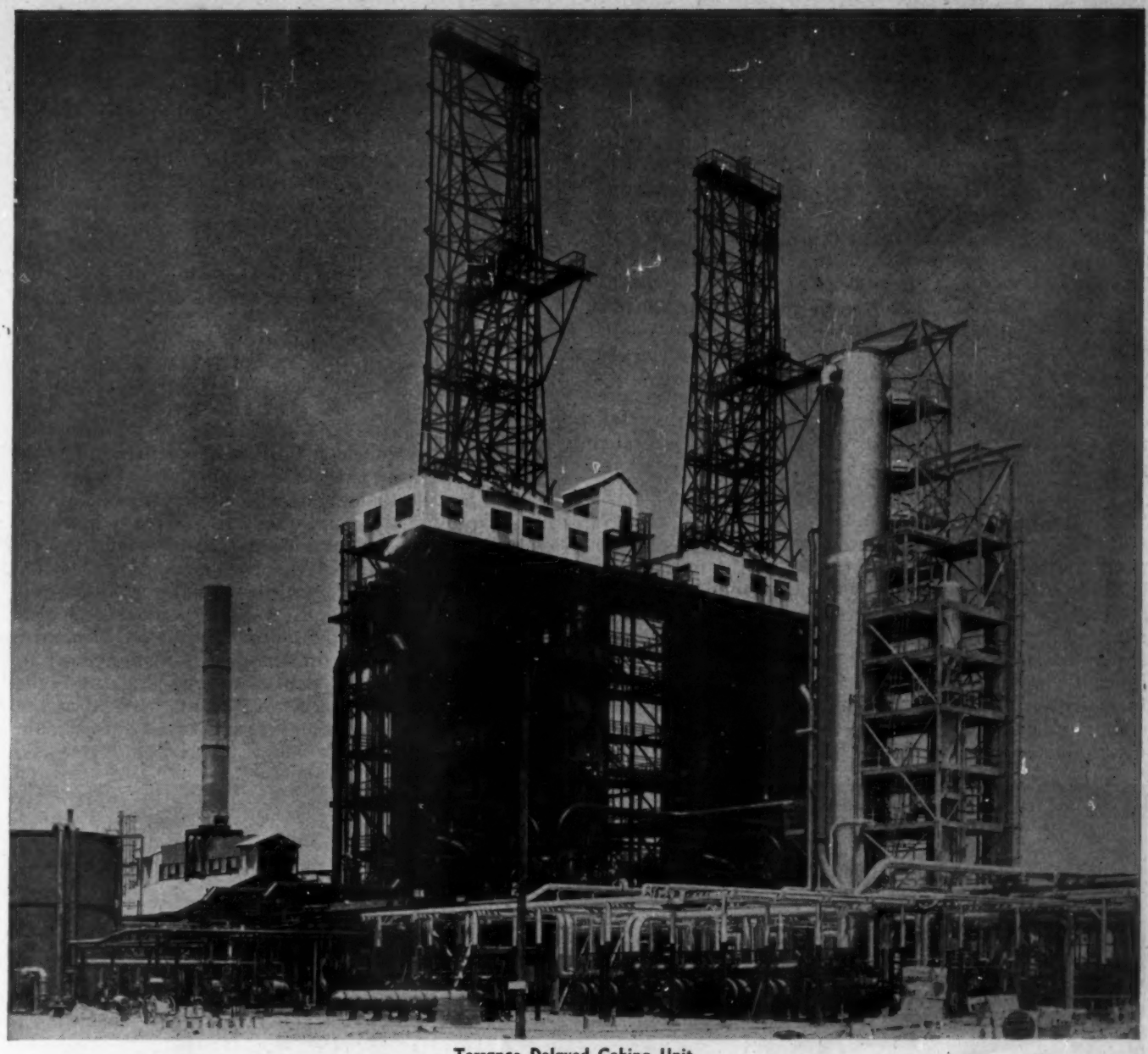
For reaming, the same tool is used, except that the bottom jet is removed and plugged off, and the four ⅜-inch nozzles are replaced with ½-inch nozzles.

The cutting tool is an hydraulically designed assembly which splits the single stream of water in the drill stem into four jet streams. Two streams are projected horizontally at the top of the tool and the other two at the bottom are directed upward at such an angle that they cut the same plane as the horizontal jets at the surface of the coke drum, or at a distance of 8½ feet from the axis of the drum. The nozzle tips in this tool are replaceable and at present ⅝-inch tips are used in the bottom nozzles, while 7/16-inch tips are used in the top.

The crusher car, which grinds the coke to a size easily pumpable by the coke pump, consists of an 11 x 14-foot hopper which feeds the coke to two 2 feet diameter by 5 feet long toothed rolls spaced



Flow Diagram, Delayed Coking Unit, General Petroleum Corporation, Torrance, California



Torrance Delayed Coking Unit

5 inches apart. The rolls are driven by a 40-horsepower motor. The crusher car is mounted on wheels and a track so that it can be positioned beneath any of the four drums. A "grizzly," fabricated of 1 x 8-inch plate, has been added near the top of the hopper to keep any large chunks from bridging over the crusher rolls or damaging them by impact.

The crushed coke and the water from the jets pass between the rolls into a sluice which leads to a coke sump. Additional sluice water is added to the ditch as needed to keep the coke and water from piling up under the crusher car. A coke slurry pump, located in a pit adjoining the coke sump, pumps the coke-water slurry through a 12-inch line to the coke storage area located some 2500 feet from the pump. The pump has clean water supplied to the case to seal the impeller. This water also serves to flush grit away from the close-clearance area between the impeller shrouds, shaft, and suction eye and the rubber lining provided in that area. The speed of rotation of the pump can be varied by means of an adjustable sheave on the motor and three sizes of sheaves on the pump. At 600 rpm the pump will discharge about 3000 gallons per minute of water over a coke outlet some 38 feet above its suction nozzle through 2500 feet of 12-inch line.

Special hydraulic-lift cars were designed to raise and lower the heavy bottom heads of the coke drums. Had only one car been provided, the track system would have been needlessly complicated to permit moving one car past the other. This complication was avoided by providing cars to be placed on the single track on both sides of the crusher car. Also, with two cars, two drum heads can be worked on at the same time. The hydraulic lifts operate on 90-pound water and are individually capable of supporting the bottom head plus the drum height of water.

This brief description of the principal process equipment should serve to clarify the following discussion of the unit operation. Additional equipment description will be included in the discussion.

Operation of the Unit

Feed Introduction

Reduced crude is charged to the bubble tower by screw-type charge pumps under flow-control which regulates the rate by positioning a by-pass valve between the pump discharge and suction. The reduced

crude is preheated by exchange successively with the light gasoil product, the medium gasoil product, the intermediate reflux stream, and the heavy gasoil product before entering the bubble tower at two points, at the top baffle tray or five baffle trays above the cracked-vapor inlet and at the second baffle tray from the bottom or two baffle trays below the cracked vapor inlet. With a preheat level of only 450° F. the feed serves as a hot reflux and care must be exercised to avoid transferring so much heat to the tower-bottom product, through a contact with the cracked vapors (the sole source of fractionating heat), as to impair the fractionation of the tower. This condition is avoided by temperature-controlling the flow of reduced crude to the top inlet so as to maintain a constant temperature on the bottom bubble tray. The excess feed is diverted by a pressure controller to the lower inlet. Any feed thus diverted also serves to subcool the tower bottom and thereby reduces the tendency to form coke in this reservoir.

Heating

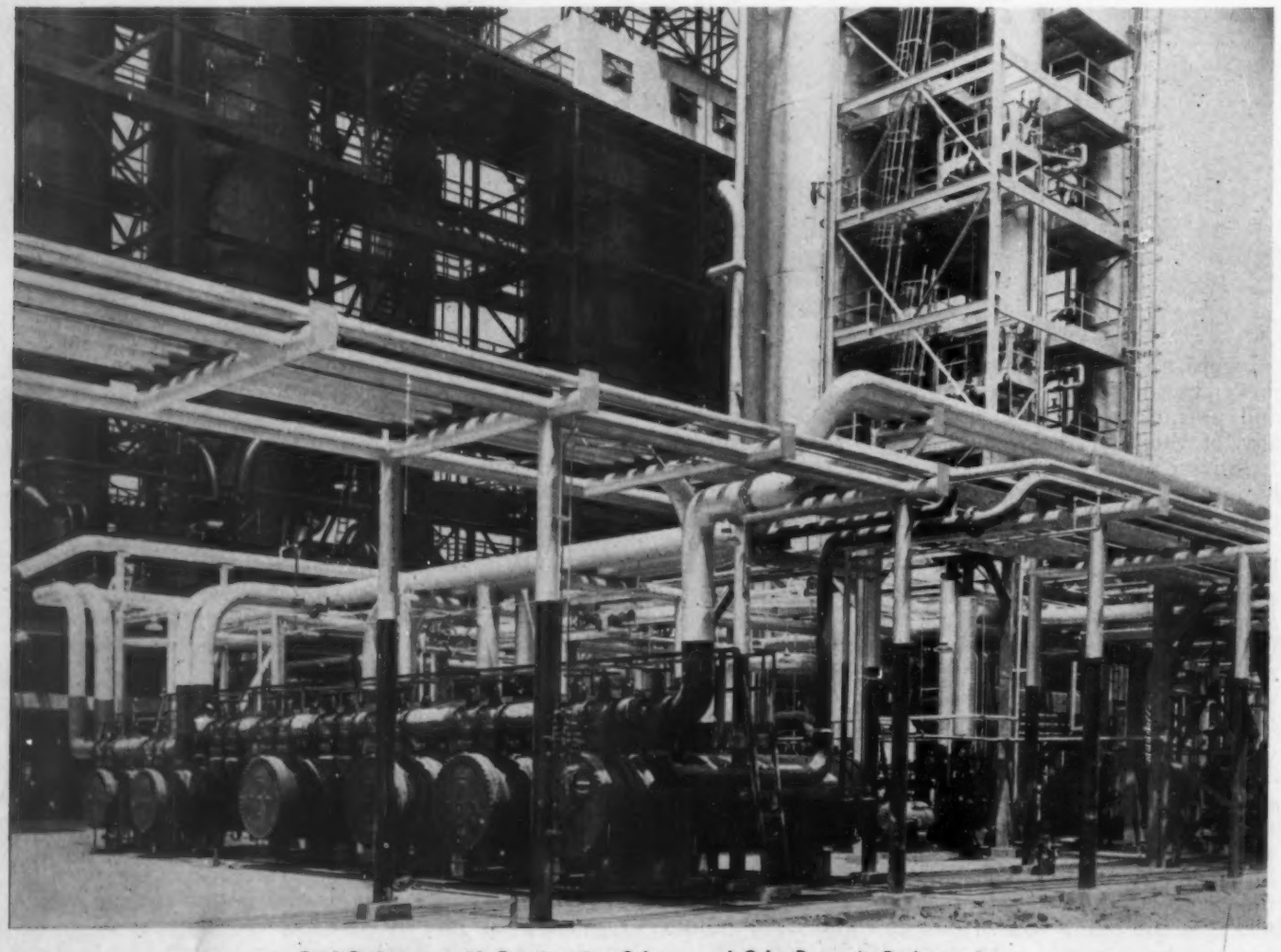
Both furnace-charge pumps take suction off the tower bottom to deliver the charge to the four parallel coils in each furnace. The radiant section of each furnace is divided into two combustion chambers containing two of the coils. The charge flow is rate controlled to each half of the furnace, and the fires in each combustion chamber are separately controlled to maintain a constant temperature at the combined outlet of each pair of coils. The two temperature-controlled outlets from each furnace join in a furnace transfer line which discharges into one of the two coke drums allocated to each furnace.

Coking

The heated mixture of vapor and liquid from the furnace enters the bottom of the coke drum. Since only vapors pass out the top of the drum, any liquid must remain in the drum until it is cracked to vapors and coke. As coke is deposited the incoming liquid and vapor must flow through the coke bed and through or into the liquid on top of the coke. With vapors and liquid continually flowing through the coking region, the coke deposited is of a highly porous nature, which facilitates subsequent handling and contributes to the quality of the product. Each drum remains on the line for 24 hours during which time approximately 250 tons are therein deposited.

Fractionation

The vapors from the two drums currently in simultaneous operation enter the bubble tower between the fifth and sixth baffle trays, numbering from the top. As previously mentioned, this stream supplies the sole source of fractionating heat to the tower. The top temperature of the tower is controlled, by means of vaporizing reflux, to pass the gasoline and lighter streams overhead. A portion of the downflow induced by the top reflux is removed from the tenth bubble tray, steam stripped, and delivered to storage, after heat exchange and cooling, as light gasoil. The remaining downflow is augmented at the fifteenth bubble tray by an intermediate reflux stream and by the additional reflux thereby induced. To



Feed Exchangers with Fractionating Columns and Coke Drums in Background

handle this additional flow the tower is enlarged at the fifteenth tray from 11 to 12½ feet in diameter. The intermediate reflux is removed from the twentieth bubble tray to be cooled by heat exchange with the fresh feed, by supplying heat to the debutanizer reboiler, and by exchange with cooling water if necessary. The intermediate reflux returned to the fifteenth tray is rate controlled. Should it be desirable to reduce the downflow on the lower bubble trays some intermediate reflux can be manually controlled to the first baffle tray. As an added means of reducing the coking tendency in the bottom two baffle trays and the tower-bottom reservoir, provision has been made to manually control some of this reflux to the seventh baffle tray if needed. The intermediate reflux draw is also used to remove the medium gasoil product which is stripped and cooled. The heavy gasoil, which is also stripped and cooled, is withdrawn from the twenty-fourth bubble tray.

The preheated fresh feed is added to the first baffle tray as required to maintain a constant temperature on the twenty-fifth or bottom bubble tray, the remaining feed being diverted to the seventh baffle tray. The tower bottoms, which include the fresh feed and recycle, are rate controlled to the furnaces. The lowest sidedraw is withdrawn at the rate required to maintain the tower-bottom level. Under this arrangement, with a constant 20,000-barrels-daily charge to the furnaces, the recycle rate is automatically determined as the difference between the fresh-feed rate and the total furnace charge.

The bubble-tower overhead is cooled and condensed in the cooling tower and is separated into gas and gasoline in the reflux accumulator. The gas flows through a back-pressure controller to the refinery central compressor station where it is compressed to the central absorption plant pressure. This controller sets the pressure for the entire system so that the pressure on the coke drums and the bubble tower are but 2 to 4 pounds higher than the 52 pounds held on the bubble-tower-reflux accumulator. The resulting pressure at the furnace outlet rides at about 75 pounds.

The stripping and coke-drum-cooling steam also are condensed in the overhead condensers and removed from the accumulator through a bottom boot. The hydrogen sulphide content is so high that this water must be steam stripped in the refinery central deodorizer before it can be delivered to sewer.

Gasoline reflux is pumped back to the top of the bubble tower under control of a temperature element located in the vapor space above bubble tray No. 2. The net gasoline make is charged to the debutanizer after heat exchange with the debutanized gasoline. The raw B-B is delivered to raw B-B storage for ultimate alky plant feed and the uncondensed vapors to the central gas plant under its own pressure. For more precise control the tower pressure is maintained by a vapor-line regulator. In order to insure a steady reflux pressure and thereby smooth out reflux flow, the accumulator pressure too is controlled within narrow limits through the use of a repressuring controller, which bypasses vapors around the condensers to maintain the accumulator pressure, and a depressuring controller which relieves the excess pressure to the gas plant.

Reboil heat is obtained from the intermediate reflux stream. To reduce the tendency to polymerize the cracked gasoline, it was decided to hold the temperature of the heating stream to 600° F. or below. Since this stream runs close to 700° F., it is first passed through the first of its four heat exchangers and is satisfactorily cooled by exchange with the fresh feed. A temperature element on the thirty-fourth tray controls the quantity of heating oil to the reboiler, by positioning a three-way control valve. After passing through the reboiler, or by-passing it, the intermediate reflux stream is further heat exchanged against the fresh feed in the remaining three exchangers and cooled if necessary before it is returned to the bubble tower.

Decoking

Drum Cycle

Operation of the coke drums requires that the furnace-transfer-line product be switched from one drum to another whenever a drum is filled with coke to the desired outage. At normal feed rates each drum is on stream coking for 24 hours and is off stream for cooling, decoking and heating up for the same period. The drum switches are so timed that one of each pair of drums comes off stream every 12 hours. Under normal conditions the cycle for one drum is about as follows:

Log of a Typical Drum Cycle

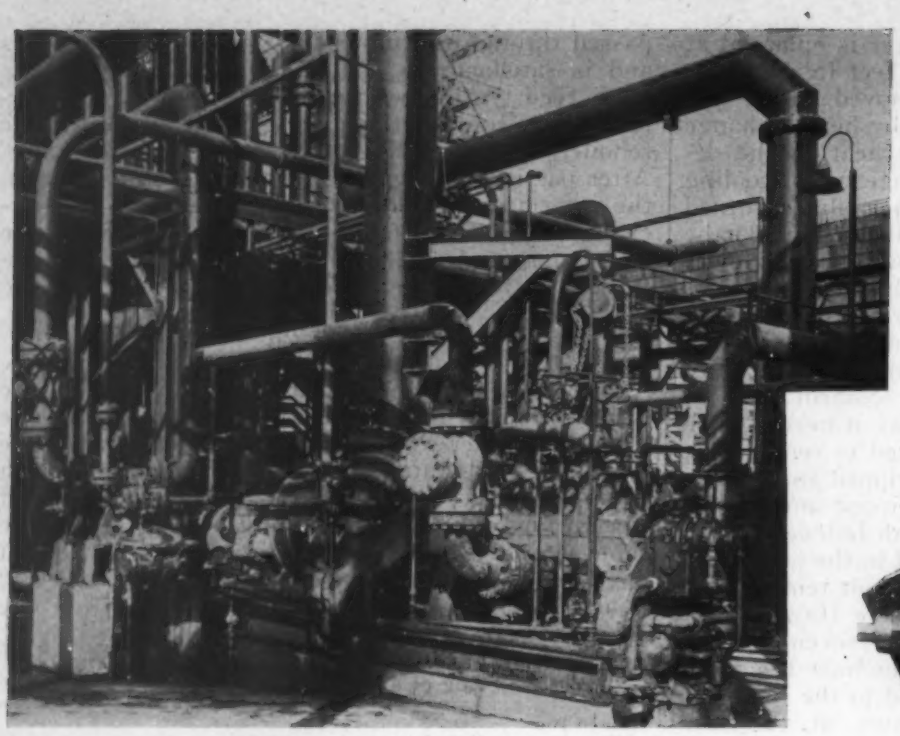
Elapsed Time (Hours)	Operation
0.0	Switch oil stream to clean drum. Steam full drum to bubble tower.
0.5	Stop steam to bubble tower, steam to blowdown.
0.75	Stop steaming, start cooling with water.
2.75	Stop cooling, start draining water, blinding and unheading.
4.0	Start decoking.
11.0	Finish decoking, start heading up and removing blinds.
12.5	Steam test.
13.0	Finish steam test.
18.0	Start heating.
24.0	Switch in oil stream from full drum.
48.0	Switch oil stream to clean drum.

The transfer line from each furnace is connected to a three-way switch valve which diverts the transfer-line product to either coke drum or through a by-pass line to the bubble tower. Before switching the oil from one drum to another, the empty drum is preheated by forcing some vapors from the on-stream drum into the top and pumping the condensate from the bottom of the drum into the bubble tower.

Blowing Down

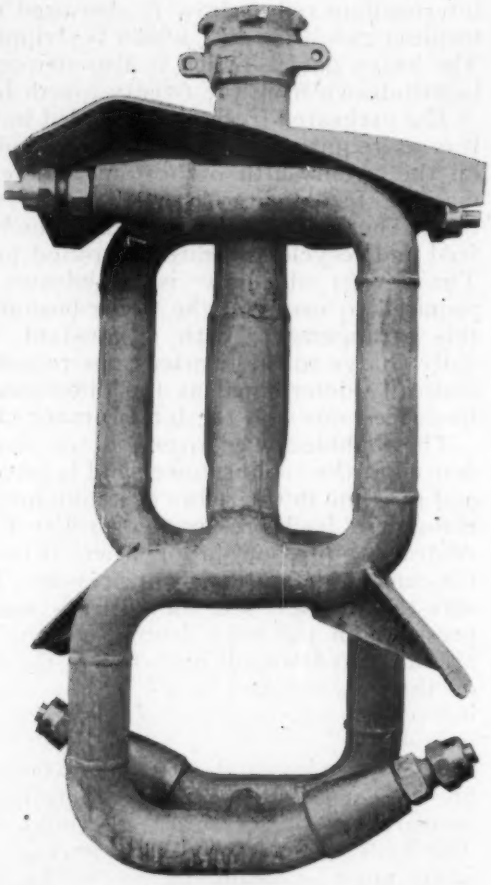
Whenever the oil stream is switched from a full to an empty drum, the full drum must be purged of volatile oils and cooled down so that decoking operations can be started. After switching out of a drum, steam is immediately cut into it, both into the transfer line to the bottom of the drum and into a steam ring just above the bottom cone. The full drum is steamed to the bubble tower for 30 minutes and is then depressured to the blowdown system. After 15 minutes steaming to the blowdown, the steam is cut out and water pumped to the steam ring.

This cooling produces considerable steam which must be handled in the blowdown system located about 2000 feet from the unit area. The blowdown line exhausts into a small tank about 6 feet below a water level maintained by a continuous circulation through the tank. The circulation is sufficient to remove the superheat of the steam and to condense any oil vapors. Some saturated steam is vented from an 80-foot stack mounted on top of the tank. The overflow from the tank runs into a skimming sump to remove any oil. The sump water is cooled by circu-



Jet water pump and turbine showing, at left, automatic by-pass valve.

Hydraulic boring tool.



Hydraulic cutting tool.

Hydraulic lift car.

Coke crusher car in operation.

lation over a 2-bay cooling tower, and a portion of the circulating water is diverted through the blowdown tank to maintain the level. The cooling tower was not designed to handle the instantaneous heat load, but rather the average heat load, the volume of water in the system acting as a flywheel or heat reservoir.

When the coke bed has been cooled to 200-300° F. the cooling water is cut out and the drum drained. It is then ready for the decoking crew to start blinding off, opening the heads, and removing the coke.

Operation of the Decoking Equipment

After a drum is cooled and drained the lines leading to the drum are blinded and the manheads removed. With the crusher car positioned under the drum, the boring is begun with the stem rotating at about 8-10 rpm. The drill is lowered as fast as possible without allowing the boring tool to actually touch the coke bed. Normally about 15 minutes are required for the high-velocity water to cut a hole through the coke. Some water leaks through the bed while drilling, but the majority is held up in the drum until the tool breaks through at the bottom. In the meantime the coke pump is started up and water alone is discharged through the coke line to the storage area until the boring operation is completed.

After the boring is completed, the reaming tool is moved slowly up and down until the hole has been enlarged to about 6 feet in diameter. This operation requires from 45 minutes to 2 hours.

After reaming, the cutting tool is installed and the final cutting is begun with the drill stem rotating at about 5-10 rpm. The coke is cut from the top of the bed and falls down through the hole into the crusher car. It is usual to lower the tool about 8 inches at a time and rotate it for 2-3 minutes before again lowering. Every 5-10 feet the jet is shut off and the drum is inspected for cleanliness. It has been found necessary to remove the coke completely on the way down since any ledges left behind for later removal frequently will fall into the crusher car and plug up the top grating or will bridge across the bottom of the drum.

Occasionally the coke is cut from the drum at such a rate that the coke pump, operating at medium speed, will not handle it. When this occurs the cutting rate is lowered by reducing the depth of cut until the excess coke is pumped away. The pump can be set to pump at a faster rate, but it is felt that a good average speed has been fixed which does not unduly delay the coking and which does not pump too much water when the cutting rate is lowered as hard coke is encountered. It is also possible to cut coke so fast that resulting large chunks cannot be handled by the crusher. With the proper care in cutting the chunks can be eliminated, but it is easily possible to cut the coke faster than the crusher can handle it. The 18-inch grating added 2 feet from the top of the crusher-car hopper protects the rolls from damage should large chunks inadvertently fall from the drum. It also retains the chunks where they can be manually broken up. When the coke is moderately hard the time required from the beginning of the boring until the drum is clean averages about 6½ hours. This corresponds to an overall coke-removal rate of about 0.7 ton per minute. Considering the time consumed in changing tools, inspecting the drum, etc., the maximum coke removal rate is probably in excess of 1 ton per minute.

When the drum is clean the transfer line and condensate line are inspected and cleaned if necessary, the steam-out valves are blasted with steam and the drum heads are replaced. After removing blinds and steam testing the drum is ready for heating prior to another 24 hours of coking service.

Operations in the Coke Storage Area

The coke-water slurry discharges onto an inclined chute the bottom of which is made of 6-mesh steel screen. The majority of the coke rolls down the screen while the water, together with all material below about 20 mesh, passes through the screen and down a solid chute to a temporary settling pond which is retained by a coke dike. A number of pipes are embedded in the dike and the water overflows through the pipes into a drainage ditch which leads to the secondary settling basin. The coke which rolls off the screen is moved by a bulldozer into piles from which it is loaded onto trucks.

This arrangement for separating the water from the coke is temporary and was devised after our original efforts along the following lines were unsuccessful. Originally it was planned that the coke would be piled in a circular dike of such proportions that the coke and water from one drum cleanout would be retained behind the dike. It was expected that the water would filter off promptly to permit moving the coke out of the way with a bulldozer to make room for the next cleanout. However, the quantity of water (1,500,000 gallons) and coke from one cleanout was so great that we had difficulty building a dike large enough to contain it. Furthermore, the fines content of the coke plugged the pores of the dike so that 3-4 days were required for the water to filter out.

A permanent coke-water-separating installation is under consideration which consists of a screen chute, a series of hoppers which can be used for loading trucks, and a primary settling basin which can be easily cleaned of coke.

To return to the water cycle, two 500,000-gallon secondary settling basins have been provided to permit the deposit removal from one while the other is in settling service. These basins are located at each end of a 3,000,000-gallon storage pond into which they overflow through submerged pipes. The pipes are so located as to leave both the settled and floating fines in the secondary settling basin. The relatively clean water from the storage pond is returned to a surge tank in the unit area by two vertical deep-well pumps. The jet pumps, the drum-cooling-water pumps, the sluice water, and the automatic make-up water to the coke-slurry pump-suction pit are all supplied from the surge tank.

Make-up water is supplied to the system from the Los Angeles Metropolitan Aqueduct System, or from the refinery wells. Tentative plans have been made to utilize the effluent water from the refinery wastewater system for this purpose.

Presently the unit is charged with 700 SSF/122 straight-run residuum from Wilmington crude at a rate of 15,000 barrels daily, which effects a recycle-to-fresh-feed ratio of 0.33/1. The 20,000 barrels daily charged to the furnaces are heated to 920° F.

The bubble tower is operated to produce gasoline with an end-point of 380° F., light gasoil with an end-point of 650° F., and medium gasoil as necessary to maintain a level in the bubble-tower-bottom reservoir. No heavy gasoil is being removed. In the stripper tower the gasoils are stripped to a flash point which makes them suitable for use as cutter stock, although at the present time the light gasoil is used

as TCC charge. Tests are being made of the feasibility of using the heavier gasoil in TCC charge.

Under the above operating conditions the following yields were obtained. Additional operating conditions have also been tabulated.

Operating Conditions and Yields

Operating Conditions	
Reduced Crude Charge	15,000 B/D
Furnace Charge	20,000 B/D
Recycle/Fresh Feed	0.33
Temperatures—Furnace Outlet	920° F.
Drum Inlet	900° F.
Drum Outlet	830° F.
Drum Pressure	56#
Viscosity of Fresh Feed	700 SSF/122
Yields	
Total Gas	14.0 Wt. %
Raw B-B	0.8 Liq. Vol. %
Debutanized Gasoline	22.5 Liq. Vol. %
Light Gas Oil	36.5 Liq. Vol. %
Medium Gas Oil	16.7 Liq. Vol. %
Coke (By difference)	19.1 Wt. %

It is rather difficult to accurately determine coke yields. Because of varying coke density, measurements by coke-drum levels do not appear to be too reliable. The above difference method includes in the coke yield any gains or losses in the process. The yields can be roughly checked by means of the weighted haulings of 500 tons per day.

It is likewise difficult to obtain representative coke samples. Samples obtained show approximately 0.25 percent ash, 9 percent volatile matter, and a screen analysis satisfactory for its intended use.

As would be expected with this type of process, the high-sulfur Wilmington residuum charge yields a gasoline with an appreciable sulfur content. This sulfur content and the olefinic content of the gasoline are sufficient to require sweetening and rerunning. Motor method clear octane numbers in the neighborhood of 70 are obtained with an octane spread of 8 or better. Lead susceptibility is fair and commensurate with the sulfur content.

Because of the desulfurizing characteristic of the TCC process, the coker gasoils have proven to be quite satisfactory as TCC charge stock despite their high sulfur contents. The 650° F.-endpoint light coker gasoil, when charged to the TCC units (bead catalyst) with a 1:1 recycle ratio, yielded 41 percent of 400° F.-endpoint debutanized gasoline. Furthermore, an octane level of 79 Motor Method clear and an octane spread of 10-12 points make this operation look quite attractive.

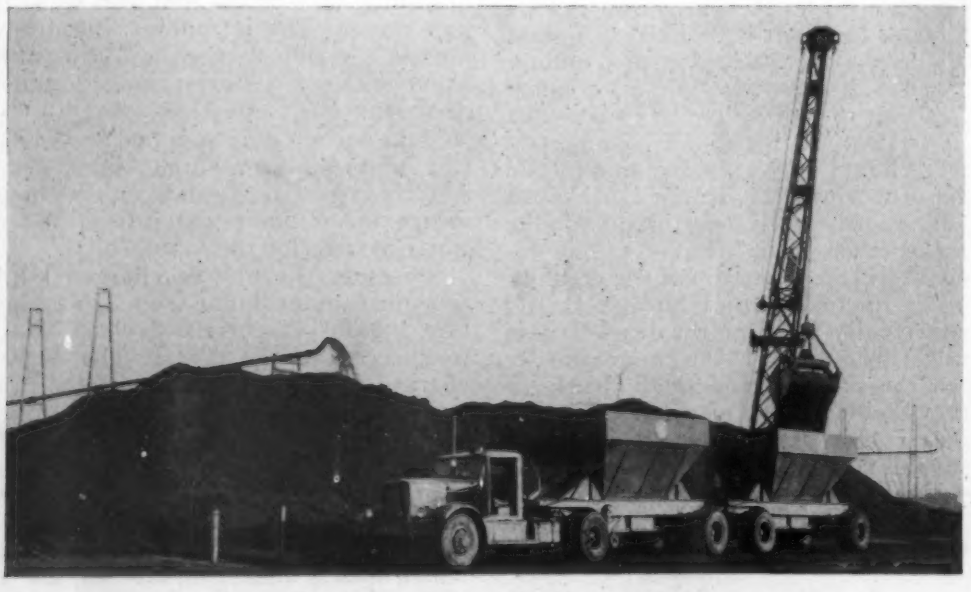
The low raw B-B yield is a result of the large gas yield from the bubble tower reflux accumulator which carries with it the bulk of the C_4 production. The total gas yield amounts to 8000-9000 thousand cubic feet per day. The dry gas after absorption amounts to some 7000 thousand cubic feet per day which is just double the requirements at the coking unit furnaces. The excess 3500 thousand cubic feet per day represents an appreciable contribution to the other refinery units' demands.

Effect of Operating Variables

Our operating time has been far too short to establish the effects of operating variables. Nevertheless, some indication of their effect has been obtained. Increased viscosity of the charge appears to shift the gasoline and gasoil production slightly toward more gas and coke. Furnace-outlet temperature changes through the range 910-920° F. also had little effect on gas and gasoline yields; however, gasoil production increased with a decrease in coke yield. Reducing the recycle ratio shifted the yields from coke to the gasoil and lighter products.

From the above indications it appears that any benefit that may be derived from a high removal of straight-run gasoil from the charge is not appreciably invalidated by lowered coker yields; yields may be varied by manipulation of the recycle ratio; and higher furnace-outlet temperatures may be feasible.

The unit is quite flexible in that it can be used either as a coker, a visbreaker, or any combination of these two. For any operation other than full coking a higher inlet to the coke drum would be used. The rate of residuum withdrawal from the bottom of the drum would determine the amount of coking permitted. In a full visbreaking operation, it is also possible to charge the fresh feed directly to the furnace along with the recycle. Under this arrangement, the residuum would be removed from the bubble-tower bottom and the recycle from the bottom draw.



Coke loading, showing coke slurry discharge.

Estimation of Properties of Solvent-Refined Products

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IN A previous paper a method has been described which permits estimation of solvent refining losses from known conditions of extraction.[1] The method is based on the applicability of infinitesimal analysis to investigations of this type and affords a convenient method of solution for a wide variety of problems both of practical and theoretical nature without requiring a large amount of experimental work. The present article has been prepared to extend the scope of the method to estimation of properties of the extracted products, a matter of considerable importance in refinery operations.

Development of mathematical expressions for correlating oil properties with conditions of extractions is complicated by the use of arbitrary scales for defining these properties in normal refinery practice. Unless the properties are expressed in fundamental units of additive nature, the resulting correlations are by necessity empirical. Further discussions are generally limited, therefore, to such fundamental units, although very satisfactory results may be secured with the artificial scales in common use by the industry.

For additive properties the following two relationships are easily derived by applying simple rules of arithmetic:

$$L = \frac{P_o - P}{P' - P} \quad (1)$$

and

$$Y = \frac{P' - P_o}{P' - P} \quad (2)$$

In these equations L represents the fractions of the charge lost to extract, Y is the yield of refined product, and P, P′ and P_o are properties of raffinate, extract and charge stock respectively. The equations are frequently employed for calculating yields from the observed specific gravities of materials involved.

Specific gravities may be considered additive properties. In extreme instances the difference between the observed and calculated gravities of petroleum blends may reach 0.65 percent or approximately 1° API. However, such variations are uncommon and are not encountered in problems connected with normal refinery practice. Direct measurements of raffinate and extract yields give results which seldom agree closer than within 1 percent on repeated extractions unless extreme precautions are observed, and discrepancies as high as 2 percent are quite common. The errors are frequently masked by the usual practice of calculating experimental data to a considerably greater accuracy than is normally warranted.

By dividing equations (1) and (2) into each other and rearranging the variables the following expression is obtained:

$$\frac{P_o - P}{L} = \frac{P' - P_o}{Y} \quad (3)$$

This shows that the difference in properties of charge stock and raffinate divided by the loss of oil to extract is equal to the difference in properties of extract and charge stock divided by raffinate yield. This function is referred to in this paper as *selectivity* of a solvent under the specific conditions of extraction employed and has been assigned the symbol X.

In solvent extractions, selectivity of the solvent changes with the quantity of solvent employed (S) and with the extraction temperature. With an increase in solvent refining losses, the difference between the properties of charge stock and raffinate also increases, suggesting that the change in selectivity with the change in severity of refining conditions may follow the same general pattern as that developed for equations involving solvent-refining losses. Validity of this assumption has been substantiated by numerous plots of available experimental data which indicate that the following relationships may be employed for calculating properties of raffinates obtained in single solvent refining:

For constant temperature and variable solvent ratio:

$$\log X = k \log S + c \quad (4)$$

For variable temperature and constant solvent ratio:

$$\log X = k'T + c' \quad (5)$$

For variable temperature and variable solvent ratio:

$$\log X = (k'' + k''' T) \log S + (c'' + c''' T) \quad (6)$$

Although equations (4), (5) and (6) were developed for additive properties of oils, such as specific gravity, which is of little interest to the refineries, they were found to apply with a considerable degree of accuracy to some other properties of oils, like viscosity index, which have little, if any, fundamental background. This is demonstrated by the experimental data presented in Tables 1, 2 and 3. The data refer to furfural, chlorex and phenol extractions of a 150 Saybolt Universal viscosity dewaxed and deasphalted Mid-Continent residuum.[2] Single-stage extractions were used in these examples, but similar results are obtained with multistage extractions as is demonstrated in sample calculations included in this paper. However, in making various interpolations or

TABLE 1

Furfural Single Stage Extractions of a 150 Saybolt Universal Viscosity Dewaxed and Deasphalted Mid-Continent Residuum

TEMPERATURE °F.	Losses Vol./vol.	Solvent Vol./vol. of Oil	VISCOSITY INDEX	
			Observed	Calculated
140	0.138	3	80.7	80.5
	0.217	6	83.8	83.9
	0.282	9	86.3	86.2
	0.338	12	87.6	87.7
160	0.171	3	81.7	81.9
	0.258	6	85.2	85.2
	0.327	9	87.8	87.3
	0.389	12	89.4	89.0
180	0.208	3	82.7	83.1
	0.305	6	86.8	86.4
	0.383	9	89.3	88.7
	0.450	12	91.2	90.4
200	0.256	3	84.6	84.5
	0.363	6	88.5	87.8
	0.448	9	91.0	90.1
	0.517	12	93.0	91.6
20	0.312	3	86.6	85.8
	0.432	6	89.8	89.3
	0.522	9	92.8	91.5
	0.597	12	94.6	93.2
240	0.384	3	88.4	87.5
	0.513	6	91.3	90.9
	0.609	9	94.4	93.1
	0.687	12	96.2	94.8
260	0.470	3	89.6	89.2
	0.610	6	92.8	92.6
	0.708	9	95.7	94.7
	0.793	12	97.3	96.5
280	0.574	3	91.0	90.9
	0.726	6	94.3	94.4
	0.828	9	96.2	96.5
	0.910	12	98.6	98.2

$$\log\left(\frac{V.I.-65}{100L}\right) = -(0.593-0.00157 \times °F) \log S + (0.735-0.0036 \times °F)$$

extrapolations the number of extractions should be constant. In extending the use of the equations to other than additive-oil properties, exercise of a considerable amount of caution is imperative and calculations should not be projected over a wide range of

TABLE 2

Chlorex Single Stage Extractions of a 150 Saybolt Universal Viscosity Dewaxed and Deasphalted Mid-Continent Residuum

TEMPERATURE °F.	Losses Vol./vol.	Solvent Vol./vol. of Oil	VISCOSITY INDEX	
			Observed	Calculated
50	0.199	3	81.5	81.3
	0.266	6	85.3	85.6
	0.315	9	88.6	88.5
	0.355	12	90.8	90.9
60	0.228	3	82.5	82.3
	0.305	6	87.7	86.7
	0.362	9	90.2	89.8
	0.409	12	92.2	92.3
70	0.263	3	84.0	83.3
	0.351	6	88.0	87.9
	0.416	9	91.5	91.1
	0.469	12	94.0	93.7
80	0.303	3	85.4	84.5
	0.405	6	89.4	89.3
	0.479	9	93.3	92.5
	0.540	12	95.5	95.2
90	0.349	3	86.3	85.8
	0.465	6	90.8	90.7
	0.551	9	94.8	94.1
	0.621	12	97.3	96.9
100	0.403	3	87.4	87.0
	0.536	6	92.5	92.2
	0.632	9	96.3	95.7
	0.713	12	98.4	98.7
110	0.463	3	88.4	88.3
	0.615	6	93.6	93.6
	0.728	9	97.8	97.4
	0.820	12	99.8	100.3

$$\log\left(\frac{V.I.-65}{100L}\right) = -(0.058+0.000515 \times °F) \log S + (0.117-0.00327 \times °F.)$$

treating conditions. Likewise, equation (3) becomes invalid.

Equations previously developed for estimating losses in single-solvent refining are of the same general type as that of equations (4), (5) and (6) with the exception that log L is substituted for log X and the value of numerical coefficients are different.[1] These equations read:

For constant temperature and variable solvent ratio:

$$\text{Log } L = a \log S + b \quad (7)$$

For variable temperature and constant solvent ratio:

$$\log L = a'T + b' \quad (8)$$

For variable temperature and variable solvent ratio:

$$\log L = (a'' + a'''T) \log S + (b'' + b'''T) \quad (9)$$

It may be noted that equations (4), (5), (7) and (8) can be easily solved by graphical methods which require very little time as compared to algebraic methods. Logarithmic paper is used for solving equations (4) and (7) and semi-logarithmic paper for solving equations (5) and (8). Algebraic methods must be applied, however, for solving equations (6) and (9).

TABLE 3

Phenol Single Stage Extractions of a 150 Saybolt Universal Viscosity Dewaxed and Deasphalted Mid-Continent Residuum

TEMPERATURE °F.	Losses Vol./vol.	Solvent Vol./vol. of Oil	VISCOSITY INDEX	
			Observed	Calculated
110	0.292	3	86.8	86.7
	0.371	6	89.2	89.4
	0.426	9	90.8	91.2
	0.471	12	92.5	92.5
120	0.308	3	86.9	87.1
	0.397	6	89.8	90.1
	0.461	9	91.8	92.0
	0.513	12	94.6	93.6
130	0.326	3	87.1	87.5
	0.427	6	90.8	89.0
	0.499	9	93.0	93.0
	0.558	12	94.7	94.4
140	0.344	3	97.3	87.8
	0.457	6	91.7	91.5
	0.540	9	94.3	93.9
	0.608	12	95.7	95.8
150	0.364	3	87.7	88.2
	0.491	6	92.8	92.3
	0.584	9	95.6	94.9
	0.661	12	97.0	96.8
160	0.385	3	88.7	88.7
	0.526	6	94.0	93.0
	0.631	9	96.6	95.9
	0.719	12	98.4	98.1
170	0.406	3	89.5	89.0
	0.564	6	94.8	93.8
	0.682	9	97.4	96.9
	0.783	12	99.5	99.4
180	0.430	3	90.4	89.6
	0.605	6	93.6	94.6
	0.740	9	98.3	98.2
	0.853	12	100.4	100.8

$$\log\left(\frac{V.I.-65}{100L}\right) = -(0.098+0.000686 \times °F) \log S + (0.096-0.0013 \times °F.)$$

Comparisons of equations (4), (5) and (6) with equations (7), (8) and (9) reveals that the same types of plots are secured irrespective of whether selectivity, i.e. the $\frac{P_o - P}{L}$ or $\frac{P' - P_o}{Y}$ functions, or simply the change in properties, i.e. $P_o - P$ or $P' - P_o$, are plotted against the other variables. Differences between the two plots are reflected merely in the values of numerical coefficients. This is easily proven by substituting values of log L from equations (7),

(8) and (9) into equations (4), (5) and (6), respectively.

For instance, equation (4) may be rewritten as follows:

$$\log X = \log \frac{P_o - P}{L} = \log (P_o - P) - \log L = k \log S + c \qquad (10)$$

Substituting value of log L from equation (7) and rearranging the variables:

$$\log (P_o - P) = (k + a) \log S + (c + b) \qquad (11)$$

Equation (11) is thus represented by a straight line plot on the logarithmic paper which is characteristic of (4) and (7).

Application of the equations shown above to the solution of practical problems is demonstrated by the following examples:

Example 1. A Coastal distillate of 370 Saybolt Universal viscosity at 100° F. was extracted with one and eight volumes of liquid sulfur dioxide at 43° F. Liquid sulfur dioxide was applied in three portions, the second and third portions being each equal to half the volume of the first portion. Raffinate yields were 90.5 percent and 74.5 percent and viscosities 326 and 274 seconds respectively. Estimate yields and viscosities of raffinates employing two and four volumes of liquid sulfur dioxide at 43° F. and using the same treating procedure.

Losses to extract corresponding to the above raffinate yields are 0.095 and 0.255 expressed as fractions of charge stock. Differences between viscosity of charge stock and viscosities of raffinates are 44 and 96 seconds. The problem is solved graphically by plotting losses against the quantities of solvent used on a logarithmic paper, drawing a straight line through the two points and finding losses corresponding to treats involving two and four volumes of solvent. The same procedure is followed for determining viscosity differences between the charge stock and raffinate. It will be found from these plots that losses corresponding to two and four volumes of solvent are 0.132 and 0.184 respectively. Raffinate yields are, therefore, 86.8 percent and 81.6 percent. Experimental data showed raffinate yields of 86.9 percent and 81.7 percent. Likewise, the interpolated viscosity differences are 57 and 74 seconds for treats employing two and four volumes of solvent respectively. The expected viscosities of raffinates are, therefore, 313 and 296 Saybolt Universal seconds at 100° F. Observed viscosities were 311 and 291 seconds.

Algebraic solution of the same problem is as follows:

Losses are determined by solving two simultaneous equations:

$$\log 0.095 = a \log 1 + b \text{ or } -1.022 = b$$
$$\log 0.255 = a \log 8 + b \text{ or } -0.593 = 0.903\,a + b$$

Therefore:

$$a = \frac{1.022 - 0.593}{0.903} = 0.475$$
$$b = -1.022$$

and

$$\log L = 0.475 \log S - 1.022$$

The corresponding equations for determining viscosity differences are:

$$\log 44 = a' \log 1 + b' \text{ or } 1.643 = b$$
$$\log 96 = a' \log 8 + b' \text{ or } 1.982 = 0.903\,a + b$$

Therefore:

$$a = \frac{1.982 - 1.643}{0.903} = 0.375$$
$$b = 1.643$$

and

$$\log (370 - V) = 0.375 \log S + 1.643$$

where V is viscosity of the raffinate.

Example 2. A Mid-Continent distillate having a viscosity-gravity constant of 0.849 was extracted with one volume of furfural at 80°F. and 140° F., using an eight-stage continuous extraction system. The yields of respective raffinates were 89.0 percent and 83.3 percent having the corresponding viscosity-gravity constants of 0.822 and 0.818. Estimate yields and viscosity-gravity constants of raffinates obtained in eight-stage extractions using one volume of solvent at 100° F. and 180° F.

Equations (8) and (5) are used in solving this problem. For graphical solution semi-logarithmic paper is employed.

Losses corresponding to 89.0 percent and 83.3 percent raffinate yields are 0.110 and 0.167 when expressed as fractions of charge stock. The corresponding differences between viscosity-gravity constants of charge stock and raffinates are 0.027 and 0.021 for the 80° F. and 140° F. treats. By plotting losses against temperature on a semi-logarithmic paper and drawing a straight line through the two points the following results are obtained which are compared with the observed values:

Temperature °F.	Losses (Vol./vol. charge) Estimated	Observed
100	0.126	0.126
180	0.221	0.222

By plotting differences of viscosity-gravity constant of charge stock and raffinates the following results are obtained:

Temperature °F.	Viscosity-Gravity Constant Estimated	Observed
100	0.821	0.821
180	0.815	0.817

Algebraic equations corresponding to the straight line plots on the semi-logarithmic paper will be found as follows:

$$\log L = 0.00303 \times °F. - 1.201$$

and

$$\log (0.849 - G) = 0.001 \times °F. - 1.649$$

where G is viscosity-gravity canstant of the raffinate.

Example 3. The following data are available regarding three-stage extractions with phenol of a 150 Saybolt Universal viscosity at 210° F. dewaxed and deasphalted Mid-Continent residuum of 65 viscosity index:

Extraction Conditions Temperature °F.	Solvent vol./vol. of oil	Raffinate Yield % Vol.	Viscosity Index
110	3	67.6	89.8
120	1	76.6	84.7
140	2	63.9	90.2
150	1	69.5	87.3

Estimate yields and viscosity indices of raffinates obtained at various temperatures and solvent ratios using three-stage counter current extractions.

This problem can be solved only algebraically using equations (9) and (6).

Losses are determined by solving the following four simultaneous equations:

$$\log (1 - 0.676) = (a'' + 110\,a''') \log 3 + (b'' + 110\,b''')$$
$$\log (1 - 0.766) = (a'' + 120\,a''') \log 1 + (b'' + 120\,b''')$$
$$\log (1 - 0.639) = (a'' + 140\,a''') \log 2 + (b'' + 140\,b''')$$
$$\log (1 - 0.695) = (a'' + 150\,a''') \log 1 + (b'' + 150\,b''')$$

or

$$-0.4895 = 0.4771\,(a'' + 110\,a''') + b'' + 110\,b'''$$
$$-0.6308 = b'' + 120\,b'''$$
$$-0.4425 = 0.301\,(a'' + 140\,a''') + b''' + 140\,b'''$$
$$-0.5157 = b'' + 150\,b'''$$

After solving these equations for the values of

TABLE 4

Raffinate yields in Three Stage Extractions of a 150 Saybolt Universal Viscosity Dewaxed and Deasphalted Mid-Continent Residuum with Phenol

TEMPERATURE °F.	Solvent Vol./vol. of Oil	LOSSES (Fraction of Charge)		RAFFINATE (Percent Volume)	
		Observed	Calculated	Observed	Calculated
110	1	0.219	0.214	78.1	78.6
	2	0.282	0.278	71.8	72.2
	3	[1]0.324	0.324	[1]67.6	67.6
	4	0.374	0.361	62.6	63.9
120	1	[1]0.234	0.234	[1]76.6	76.6
	2	0.303	0.303	69.7	69.7
	3	0.347	0.353	65.3	64.7
	4	0.398	0.394	60.2	60.6
130	1	0.251	0.256	74.9	74.4
	2	0.330	0.330	67.0	67.0
	3	0.374	0.384	62.6	61.6
	4	0.427	0.427	57.3	57.3
140	1	0.274	0.280	72.6	72.0
	2	[1]0.361	0.361	[1]63.9	63.9
	3	0.407	0.418	59.3	58.2
	4	0.454	0.465	54.6	53.5
150	1	[1]0.305	0.305	[1]69.5	69.5
	2	0.398	0.393	60.2	60.7
	3	0.446	0.454	55.4	54.6
	4	0.506	0.504	49.4	49.6
160	1	0.336	0.334	66.4	66.6
	2	0.436	0.427	56.4	57.3
	3	0.490	0.493	51.0	50.7
	4	0.552	0.547	44.8	45.3
170	1	0.369	0.365	63.1	63.5
	2	0.476	0.466	52.4	53.4
	3	0.536	0.537	46.4	46.3
	4	0.600	0.596	40.0	40.4
180	1	0.404	0.398	59.6	60.2
	2	0.517	0.506	48.3	49.4
	3	0.583	0.583	41.7	41.7
	4	0.651	0.646	34.9	35.4

[1] Reference points for equation.
log L=(0.421—0.0004 x °F) log S—(1.091—0.00384 x °F.)

numerical constants and substituting these values into equation (9) the following expression is secured:

$$\log L = (0.421 - 0.0004 \times {}^\circ F) \log S - (1.091 - 0.00384 \times {}^\circ F)$$

Table 4 compares results obtained from this equation with the experimental data.

Viscosity indices are determined by solving the following equations:

$$\log\left(\frac{89.8-65.0}{100}\right) = (k'' + 110\,k''')\log 3 + (c'' + 110\,c''')$$

$$\log\left(\frac{84.7-65.0}{100}\right) = (k'' + 120\,k''')\log 1 + (c'' + 120\,c''')$$

$$\log\left(\frac{90.2-65.0}{100}\right) = (k'' + 140\,k''')\log 2 + (c'' + 140\,c''')$$

$$\log\left(\frac{87.3-65.0}{100}\right) = (k'' + 150\,k''')\log 1 + (c'' + 150\,c''')$$

or

$$-0.605 = 0.477\,(k'' + 110\,k''') + c'' + 110\,c'''$$
$$-0.705 = c'' + 120\,c'''$$
$$-0.599 = 0.301\,(k'' + 140\,k''') + c'' + 140\,c'''$$
$$-0.652 = c'' + 150\,c'''$$

Numerical values of constants obtained from these equations are substituted in equation (6) leading to the final expression:

$$\log\left(\frac{V.I.-65.0}{100}\right) = (0.302 - 0.00049 \times {}^\circ F)\log S - (0.921 - 0.0018 \times {}^\circ F)$$

The 100 denominator is used in this and other equations for convenience in arithmetical calculations only.

Table 5 compares calculated and observed viscosity indices of the stock.

The above sample calculations involve several oil properties in order to demonstrate the general usefulness of the proposed method. It should be emphasized that interpolations and, particularly, extrapolations should not be carried out to an extreme. Small experimental errors involved in determining the basic points are also magnified by extending the curves over wide ranges of temperatures and solvent-extraction conditions.

The expression $\frac{P_o - P}{L}$ in the equation above has been defined as the measure of *selectivity* of a solvent. Similarly the expression $\frac{L}{S}$ defines the *solvent power* of a solvent, i.e. the quantity of oil which the solvent is capable of holding in solution. Product of selectivity and solvent power or $\frac{P_o - P}{S}$ should be referred to as the *refining power* of a solvent showing improvement in raffinate properties with the quantity of solvent employed irrespective of losses. The refining power of solvents is a highly important characteristic because it is a measure of the quantity of solvent needed for securing the desired improvement in raffinate properties. Although losses do not enter into the definition of solvent power, this is justified in view of the application of counter-current extraction methods in commercial practice. These methods permit securing equal degrees of improvement in raffinate properties, particularly their viscosity indices, for equal oil losses to the extract provided the number of extraction stages is adjusted according to selectivity characteristics of individual solvents.

Selectivity of solvents at various temperatures and for different solvent to oil ratios is calculated directly from equations (4), (5) or (6). Solvent power is

TABLE 5

Viscosity Indices of Raffinates from Three Stage Extractions with Phenol of a 150 Saybolt Universal Viscosity Dewaxed and Deasphalted Mid-Continent Residuum

TEMPERATURE °F	Solvent Vol./vol. of Oil	VISCOSITY INDEX	
		Observed	Calculated
110	1	84.0	83.9
	2	87.2	87.5
	3	[1]89.8	89.8
	4	92.0	91.7
120	1	[1]84.7	84.7
	2	88.4	88.3
	3	90.3	90.8
	4	93.0	92.6
130	1	85.5	85.6
	2	89.3	89.3
	3	91.2	91.7
	4	94.2	93.6
140	1	86.3	86.4
	2	[1]90.2	90.2
	3	92.2	92.7
	4	95.5	94.6
150	1	[1]87.3	87.3
	2	91.6	91.2
	3	93.4	93.7
	4	97.3	95.7
160	1	89.0	88.3
	2	92.4	92.2
	3	94.6	94.8
	4	98.3	96.8
170	1	90.0	89.3
	2	93.2	93.3
	3	95.5	95.8
	4	99.0	97.9
180	1	90.7	90.3
	2	94.0	94.3
	3	96.7	97.0
	4	99.5	99.0

[1] Reference points for equation.

$$\log\left(\frac{V.I.-65}{100}\right) = (0.302 - 0.00049 \times {}^\circ F)\log S - (0.921 - 0.0018 \times {}^\circ F.)$$

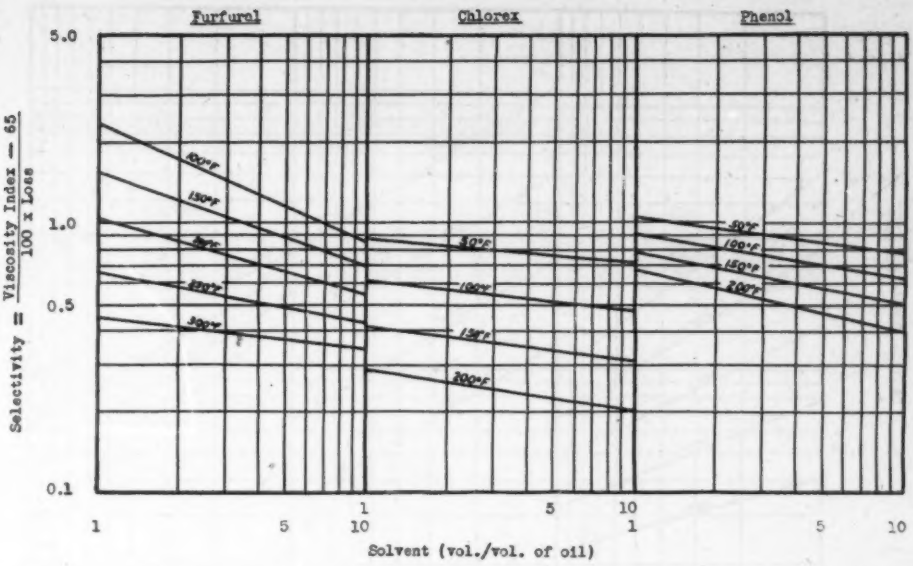

FIGURE 1
Selectivity of Solvents.

(Single-stage extractions of deasphalted Mid-Continent residuum of 150 Saybolt Universal viscosity at 210° F. and 65 viscosity.)

determined by employing equations (7), (8) or (9) in a slightly modified form, and refining power by summation of the corresponding equations. This is demonstrated by the following examples involving equations (6) and (9).

Selectivity $\frac{P_o - P}{L} = X$

$$\log X = (k'' + k'''T) \log S + (c'' + c'''T) \qquad (12)$$

The equation is identical with equation (6)

Solvent Power $\frac{L}{S} = Z$

$$\log Z = (m + nT) \log S + (p + q'T) \qquad (13)$$

In this equation coefficient $m = a'' - 1$ of equation (9) while coefficient n, p and q are identical with the coefficients a''', b'' and b''' respectively.

Refining Power $\frac{P_o - P}{S} = R$

$$\log R = (m' + n'T) \log S + (p' + q'T) \qquad (14)$$

In this equation $m' = k'' + m$; $n' = k''' + n$; $p' = c'' + p$; and $q' = c''' + q$ of equations (12) and (13).

In the above equations solvent characteristics are expressed in terms of the quantities of solvents used and temperature. However, these two variables are interdependent with solvent losses and assignment of numerical values to any two of these three variables automatically determines the value of the third one. For this reason equations (12), (13) and (14) may be presented in any one of the three forms.

Application of expressions relating to solvent characteristics of solvents to practical problems is demonstrated by the following examples involving extractions of a dewaxed and deasphalted Mid-Continent residuum. The basic equations from which the expressions below are derived have been presented in this and in a previous publication:[1]

Furfural extractions

$$\log X = -(0.593 - 0.00157 \times °F) \log S + (0.735 - 0.0036 \times °F)$$

$$\log Z = -(0.053 + 0.00220 \times °F) \log S - (1.924 - 0.00545 \times °F)$$

$$\log R = -(0.646 + 0.00063 \times °F) \log S - (1.189 - 0.00185 \times °F)$$

Chlorex extractions

$$\log X = -(0.058 + 0.000515 \times °F) \log S + (0.117 - 0.00327 \times °F)$$

$$\log Z = -(0.574 + 0.00014 \times °F) \log S - (1.212 - 0.0062 \times °F)$$

$$\log R = -(0.632 + 0.000665 \times °F) \log S - (1.095 - 0.00293 \times °F)$$

Phenol extractions

$$\log X = -(0.098 + 0.000686 \times °F) \log S - (0.096 - 0.0013 \times °F)$$

$$\log Z = -(0.885 - 0.0021 \times °F) \log S - (0.854 - 0.0014 \times °F)$$

$$\log R = -(0.983 - 0.001414 \times °F) \log S - (0.758 - 0.0001 \times °F)$$

The straight lines represented by these equations on a logarithmic paper are plotted in Figures 1, 2 and 3 for various temperature levels and for quantities of solvents ranging from one to ten per volume of charge stock.

The following points were calculated from the above equations and used in constructing Figures 1, 2 and 3:

Temp. °F.	Solvent Vol./Vol. Oil	Selectivity (X)			Solvent Powder (Z)			Refining Powder (R)		
		Furfural	Chlorex	Phenol	Furfural	Chlorex	Phenol	Furfural	Chlorex	Phenol
50	1	.....	0.897	1.070	.....	0.125	0.164	.....	3.113	0.177
50	10	.....	0.733	0.792	.....	0.033	0.027	.....	0.024	0.022
100	1	2.37	0.617	0.925	0.042	0.256	0.193	0.099	0.158	0.179
100	10	0.87	0.479	0.629	0.022	0.066	0.041	0.019	0.032	0.026
150	1	1.57	0.423	0.796	0.078	0.522	0.227	0.122	0.221	0.181
150	10	0.69	0.310	0.501	0.032	0.133	0.061	0.022	0.041	0.031
200	1	1.04	0.290	0.685	0.146	1.070	0.267	0.152	0.310	0.183
200	10	0.54	0.200	0.399	0.047	0.267	0.091	0.026	0.054	0.037
250	1	0.68	.....	.....	0.274	.....	.....	0.188	.....	.....
250	10	0.43	.....	.....	0.068	.....	.....	0.030	.....	.....
300	1	0.45	.....	.....	0.514	.....	.....	0.232	.....	.....
300	10	0.34	.....	.....	0.100	.....	.....	0.034	.....	.....

Highest numerical values obtained from the above expressions are indicative of the desirable solvent characteristics of solvents. High selectivity shows that the number of extraction stages may be reduced to a minimum without discarding appreciable quantities of valuable oil constituents to the extract. High solvent power demonstrates ability of a solvent to dissolve the undesirable oil constituents. This characteristic is very important because it depends entirely on the nature and inherent characteristics of solvents and is changed only to a relatively minor extent by increasing the number of extraction stages but varies widely with the temperature. High refining power shows that a given improvement in oil characteristics may be secured with a minimum solvent circulation under the selected set of extracting conditions including the number of extraction stages employed.

Further discussions refer to the oil-solvent systems selected for the present studies and should not be generalized because the results may vary with the nature of available charge stocks which determines

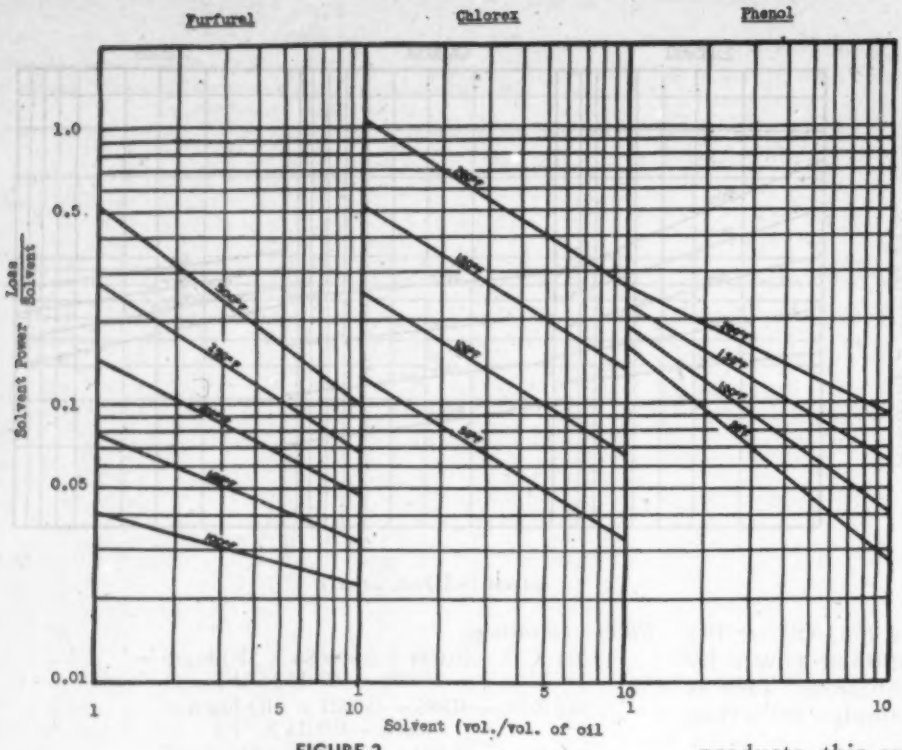

FIGURE 2

Solvent Power of Solvents.

(Single-stage extractions of deasphalted Mid-Continent residuum of 150 Saybolt Universal viscosity at 210° F. and 65 viscosity index.)

economical value of various solvents for the individual refineries. These discussions are presented, therefore, for illustrative purposes only.

The data plotted in Figures 1, 2 and 3 demonstrate that by making suitable adjustments for the temperature differences all three solvents fall within approximately the same ranges of selectivity and solvent power. However, their refining power shows considerable variations. While the temperature differentials are rather large for furfural and chlorex, a change in extraction temperature under similar conditions has only a minor effect on phenol extractions. This is particularly true for low solvent ratios such as are employed in commercial plant operations. Significance of these differences may be visualized from the following general considerations.

In solvent refining properties and yields of raffinates are usually controlled by the extraction temperature and quantity of solvent used. In properly designed commercial equipment sufficient number of stages is provided in order to avoid the loss in selectivity which occurs at elevated temperatures. The plant operator has, therefore, two variables at his disposal for adjusting the quality of raffinates without incurring unduly high oil losses to the extract. These variables are temperature and solvent to oil ratio. It is thus possible to produce a variety of raffinates by keeping the solvent ratio within rather narrow limits and secure the desired performance by properly selecting the extraction temperature. This is frequently of much importance because increased solvent circulation reduces the unit capacity and raises the cost of refining per barrel of raffinate produced. If the refining power shows only a slight change with the temperature, the quality of raffinate is practically fixed with the quantity of solvent employed and solvent circulation is the sole variable at the disposal of the operator for securing the variety of products which is demanded from him.

The above discussion does not take into consideratin special methods which were developed and are used commercially for improving flexibility of solvent-extraction operation. Whenever a certain solvent lacks some desirable property, preventing its successful application over the desired range of products, this can be remedied by changing the solvent composition, which is changed by employing mixtures of solvents, like liquid sulfur dioxide and benzol, the first solvent supplying the desired selectivity and the second supplying the solvent power to the blend. The same effect may be accomplished by injection of water which is an established feature of the phenol process. As a result of these various modifications the required flexibility of operation is obtained and sometimes even improved over that observed with other solvents.

The present article has been confined to single-solvent extractions. The same principles may be extended, however, to other oil-solvent systems by applying the suggested method of handling scanty data which are normally available to their best advantage.

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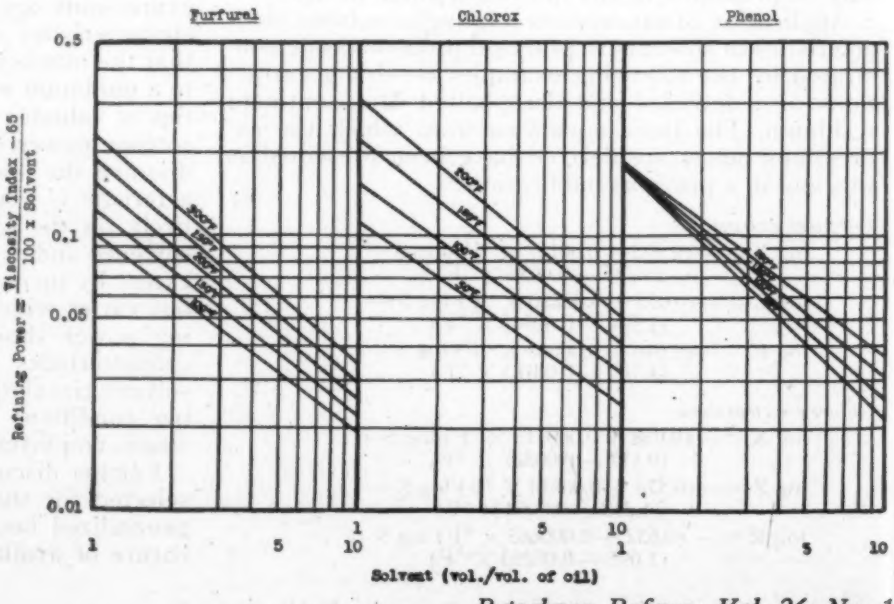

FIGURE 3

Refining Power of Solvents.

(Single-stage extractions of deasphalted Mid-Continent residuum of 150 Saybolt Universal viscosity at 210° F. and 65 viscosity index.)

Some Practical Applications of

Flue Gas Recirculation

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USE of flue gas recirculation to temper the gases of combustion ahead of separately-fired superheaters, air heaters, and process equipment is not new. There is novelty however in many modern applications, and particularly in the major details which can make or mar the success of the entire installation. For this reason a description of the design and operation of the high-temperature installation at Neches Butane Products Company may be of interest.

As a brief review of the background for gas recirculation,—the temperature of the gases from direct combustion with normal excess air is ordinarily too high for direct application to heat exchangers, in which the heat-absorbing medium is steam or gas, unless tremendously high mass flows with their costly pressure losses or expensive alloys, or both, are resorted to. Even though such special means may justify themselves theoretically in very exceptional cases, the margin of safety against overheating of metal parts is so slim that a very small lapse from practically perfect operation usually will result in severe damage to the equipment. Gas recirculation reduces the temperatures of the gases entering the heat exchanger to the point where sensible design conditions may be safely projected into actual operation, carried out with reasonable care and under simple control limitations.

Where the efficiency of the unit is unimportant, the products of combustion may be cooled by admitting air to the gases before they enter the heat exchanger. With this method of controlling the gas temperature, the heat transfer can be reduced and the tube-metal temperature in the heat exchanger can be maintained at safe levels. It is readily apparent, however, that this is an inefficient method of operation since the stack losses are increased in almost direct proportion to the weight of cooling air admitted.

If, instead of air, a portion of the relatively cool gases leaving the heat exchanger is recirculated to the furnace and mixed with the products of combustion, the advantages of the reduced gas temperatures entering the exchanger may be obtained without an appreciable loss in efficiency. By proper control of the recirculated gases:

1. A lower furnace temperature is maintained, which results in lower furnace maintenance.
2. The gas temperature entering the heat exchanger is sufficiently low to permit control of metal temperatures.
3. In spite of the reduction in heat transfer desired and accomplished at the entrance to the heat exchanger, the average absorption is decreased very little because of the higher mass flow of the gases.
4. The gas weight to the stack or supplementary heat-transfer equipment is that corresponding to the low excess air required for efficient combustion, which results in a minimum stack loss.
5. The gases used for recirculation will have sufficient sensible heat removed from them to permit efficient handling by a fan, and easy introduction through simple ports and dampers.
6. Some additional work will be expended by the fan required for recirculating the gases.

PART of the concluding paragraph of this paper provided this summary and estimate of further applications of flue-gas recirculation in refinery and petro-chemical processing:

"Flue-gas recirculation has many applications. As described here it is excellently suited for separately-fired superheaters and air heaters. It is applicable also to special process equipment such as benzene superheaters in the plastic industry and for oil heaters in the petroleum refining industry. In the chemical industry the temperature of chemical-bearing gases may be controlled by recirculation in the cooling-down process. In the power field where space conditions prevent the use of sufficient radiant-heat-absorbing surface to cool the gases below the fusion point of the ash, flue-gas recirculation may be used to cool gases and prevent slagging trouble. Finally flue-gas recirculation may play an important part in certain types of gas-turbine applications."

A contribution of the Fuels Division of The American Society of Mechanical Engineers, this paper was presented at its annual meeting in New York, December 6, 1946.

The design of a recirculated-flue-gas unit is quite simple. It consists primarily of:

1. Forced-draft fan and combustion equipment, similar to that for a once-through unit.
2. A combustion chamber and mixing furnace.
3. Heat-absorption equipment.
4. Combination recirculation and induced-draft fan.
5. Recirculated-gas ducts and ports.
6. Dampers for controlling the forced draft, recirculated gases, and the induced or stack draft.
7. Air heater and automatic control of the fuel and dampers (optional).

A comparison of two practical methods of controlling the temperature of the gases entering the heat exchanger (by regulation of excess air and by recirculation of flue gases) is listed in Table 1. This comparison is made on the basis of the Neches Butane installation which will be described below. It will be noted that the gas recirculation arrangement is ten percentage points better in efficiency and requires less fan power than the unit using increased excess air. In addition the air heater is only 40 per cent as large as for the arrangement using increased excess air, which means a substantial saving in first cost.

The arrangement of the separately-fired super-

TABLE 1

METHOD	Gas Recirculation	Increased Excess Air
Furnace	Same	Same
Superheater	Same	Same
Air heater—sq. ft.	7,360	19,000
Efficiency of unit—percent	80.3	70.8
Gas weight leaving superheater, lb./hr.	230,200	242,600
Excess air at superheater outlet, percent	21	194
Air weight leaving air heater, lb./hr.	74,400	225,500
Steam flow, lb./hr.	142,500	142,500
Press. at superheater inlet, psi	100	100
Press. at superheater outlet, psi	50	50
Temp. at superheater outlet, °F.	1400	1400
Gas temp. entering superheater, °F.	1860	1860
Gas temp. leaving superheater, °F.	690	690
Gas temp. leaving air heater, °F.	390	390
Draft loss through unit, in. water	6.2	6.2
Air resistance through unit, in. water	6.7	6.7
Forced-draft fan, BHP	29	86
Induced-draft fan, BHP	169	140
Total, BHP	198	226
Gas weight recirculated, lb./hr.	139,200	.....
Gas weight recirculated, percent	169	.....
Tempering air, lb./hr.	.....	140,900
Tempering air, percent	.....	167
Induced-draft fan location	Before A.H.	After A.H.

ments of the synthetic-rubber industry, six separately fired superheaters, each designed for a capacity of 142,500 pounds of steam per hour at 1400° F. and 50 psi at the superheater outlet were supplied for the butadiene plant at Neches. To simplify the design and to conserve material, a superheater and a waste-heat boiler or cooler were installed adjacent to a butylene heater to form an integrated unit.

Separately-Fired Superheater

The arrangement of this separately-fired superheater (as shown in Figure 1) consists of a combustion chamber in which natural gas is burned and in which recirculated gases are introduced into the mixing chamber through the ports located at floor level, a solid bridge wall for mixing the new gases with the recirculated gases, and a checker o lter wall to assure proper distribution of the gases entering the tube bank and to protect the tube bank from the radiant heat of the furnace. The combination recirculation-and induced-draft fan is located at the outlet of the superheater tube bank. In this particular case

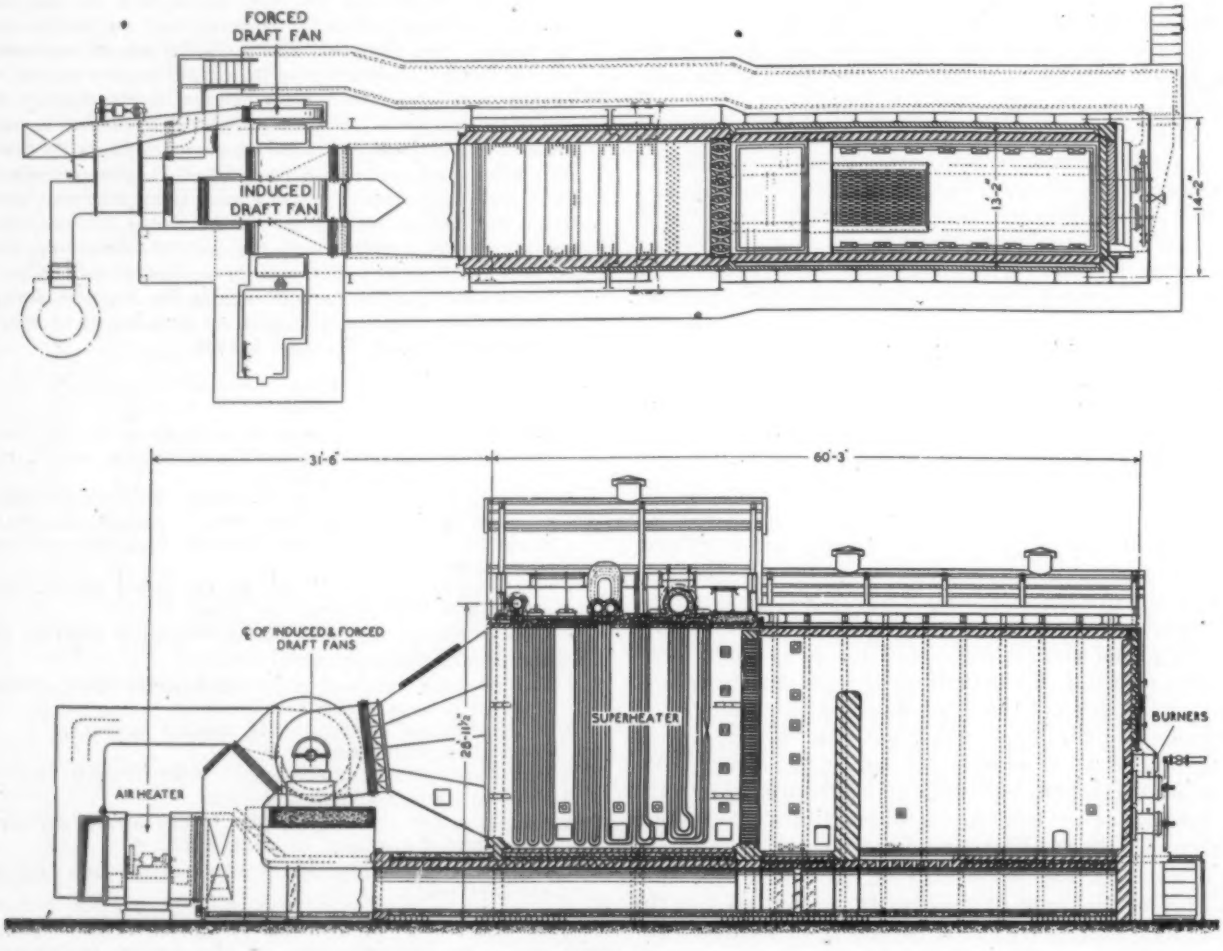

FIGURE 1

heaters at the Neches Butane Products Company located at Port Neches, Texas, utilizing flue-gas recirculation, is shown on Figure 1. The dehydrogenation of butylene to butadiene requires steam as a carrier. Within reasonable limits the higher the steam temperature the shorter the time required for the dehydrogenation to take place. In other words, steam temperature has a direct bearing on capacity.

In order to help meet the wartime capacity require- the forced-draft fan is driven from a common shaft with the recirculation-induced-draft fan. The forced-draft fan discharges air through the air side of the air heater and then through a duct to the burners for the combustion of the fuel. The recirculation-induced-draft fan discharges the flue gas into a duct which divides into two sections, one section returning a portion of the gases through the duct underneath the floor and introducing it through the several ports into

the furnace, and the second section of the duct discharging the remaining gases through the air heater and on to the stack. While the recirculated gases are nothing more than a controlled eddy they serve the most useful purpose of leveling off the temperature of

In operation, the amount of flue gas recirculation necessary to keep the tube temperatures within the safe limits for this alloy is established. After this is worked out for the maximum load there is no further need for altering the setting of the dampers. The

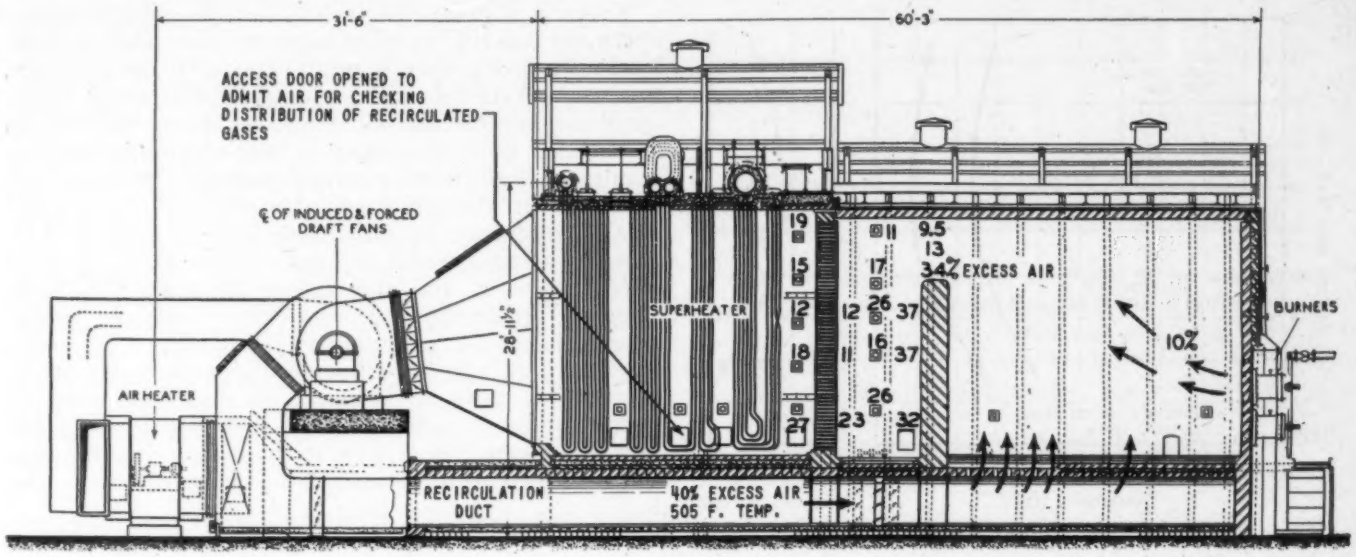

FIGURE 2

the gases entering the heat exchanger, distributing the heat-transfer load more evenly over the entire tube bank, as well as cooling the refractory furnace floor and furnace walls.

As a guide in the operation of these superheaters, thermocouples were installed in the gas stream between the filter baffle and the first row of tubes. Special thermocouples were installed to measure the metal temperature and also the steam temperature of a number of the tubes at the outlet end.

TABLE 2

Steam pressure to superheater	100 lb./sq. in.
Steam pressure from superheater	22 lb./sq. in.
Steam flow	143,600 lb./hr.
Steam temp. leaving superheater	1326 °F.
Temperatures:	
Gases entering superheater (High-velocity thermocouple)	1685 °F. avg. of 12 TC
Gases leaving superheater	725 °F. avg. of 8 TC
Gases leaving air heater	481 °F.
Air entering air heater	65 °F.
Air leaving air heater	613 °F.
Steam temperature entering Superheater	340 °F.
Steam temperature at cross-over heads	943 °F. avg. of 8 TC
Steam temperature leaving superheater	1326 °F.
Tube temperature front row	1374 °F. avg. of 14 TC
Tube temperature at superheater outlet	1335 °F. avg. of 10 TC
Drafts:	
Entering air heater	+3.5 in. water
Leaving air heater	+1.0
Recirculating gas:	
Right side wall duct	+2.5
Center recirculating duct	+0.9
Left side wall duct	+2.5
Furnace	−0.1
Ahead of filter wall	−0.35
After filter wall	−2.20
Leaving superheater	−5.5
ID fan to recirculating ducts	+2.1
Excess air at superheater outlet	17 percent
Damper adjustments:	
1—Main recirculation damper	Wide open
2—Right side wall duct damper	Wide open
3—Center recirculation damper	60 percent open
4—Left side wall duct	Wide open
5—Damper nearest burner (A)	100 percent open
Damper nearest burner (B)	75 percent open
Damper nearest burner (C)	20 percent open
Damper nearest burner (D)	0 percent open

The average refractory temperature at burner zone was about 2700 °F. as measured by optical pyrometer. The calculated heat liberation is 19,200 Btu/ct. ft. and the heat available (including preheated air and recirculated gases) is 24,800 Btu/ct. ft.

forced draft is regulated in accordance with the air required for the combustion of the fuel. Induced draft is regulated to provide the proper furnace draft.

The actual performance of the superheaters at approximately design conditions is shown in Table 2. The operation is guided by the requirements to obtain the best results for the dehydrogenation of butylene, rather than by strict adherence to the design conditions of the superheater. Therefore, the units are normally base-loaded at about 165,000 pounds of steam per hour with 1350° F. final temperature and 25 psi at the superheater outlet. Because of the complete control of the furnace and metal temperatures gained by the use of flue-gas recirculation the performance of these superheaters has been excellent. They have been in service for approximately three years at this high-steam-temperature level and recent inspection shows that the tubes, which are 2-inch OD Croloy 18-8 columbium stabilized steel, are in excellent shape with no signs of oxidation or creep.

The thoroughness with which the recirculated gases are mixed with the gases of combustion is most important since this is one of the main criterions of tube-metal temperature. The effectiveness of the arrangement used on the superheaters at Neches consisting of the solid bridge wall followed by the filter baffle wall is illustrated on Figures 2, 3, 4, and 5.

Figure 2 shows the cross-sectional elevation of the unit. The large access door located at the mid-point of the tube bank was opened during the test when these data were obtained in order to raise the excess air of the recirculated gases somewhat above the excess air in the combustion zone. This made it possible to trace the recirculated gases through the unit by checking the excess air as well as the gas temperature. The spot readings of excess air are shown on Figure 2.

Figure 3 shows a plot of the gas temperature and excess air immediately ahead of the tube bank. The flue-gas temperatures entering the superheater were measured with bare thermocouples and also with a

high-velocity thermocouple. Because the high-velocity thermocouple is shielded from the cooler heat-absorbing surfaces and aspirates the flue gases over it at high velocity it reads considerably higher than the bare thermocouples used as an operating guide, and provides the temperature-correction factor which

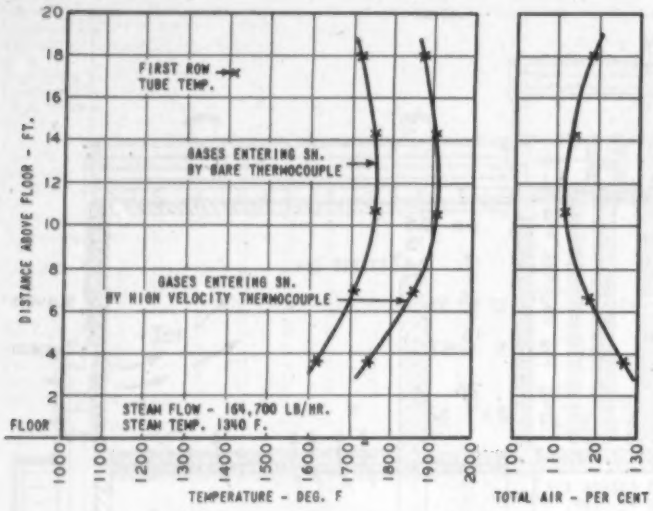
FIRST ROW TUBE TEMP.
GASES ENTERING SH. BY BARE THERMOCOUPLE
GASES ENTERING SH. BY HIGH VELOCITY THERMOCOUPLE
STEAM FLOW - 164,700 LB/HR.
STEAM TEMP. 1340 F.
FLOOR
DISTANCE ABOVE FLOOR - FT.
TEMPERATURE - DEG. F
TOTAL AIR - PER CENT

FIGURE 3

must be applied to these bare thermocouples. It will be noted that the gas temperatures and excess airs are quite uniform, with the exception of the lowest elevation which undoubtedly is influenced by a certain amount of air infiltration.

Figure 4 shows a contour plot of the gas temperatures across the width and the height of the unit at the entrance to the superheater as measured by the high-velocity thermocouple. This shows a total variation in gas temperature of 250 degrees at this location with the temperatures around the boundary surface being somewhat cooler, as it usually is.

Figure 5 shows the temperature of the individual superheater elements across the width of the unit. The top curve gives the metal temperatures taken at a point 17 feet above the floor and in the gas stream,

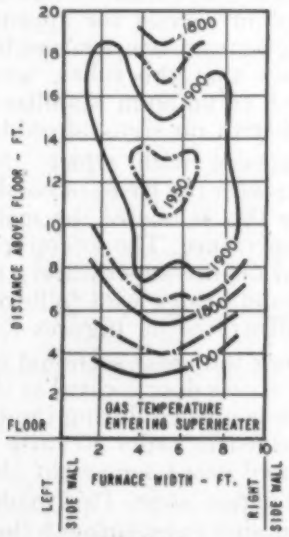

FIGURE 4

and represents probably the hottest metal temperature that exists in the unit. The second curve was obtained on the tubes out of the path of the gases and represents steam temperatures in the individual tubes. The bottom curve illustrates the steam temperatures at the cross-over headers where the steam temperatures are balanced by crisscrossing of the connecting tubes to counteract any possible effect of unbalanced gas flow through the superheater. It will be noted that the steam and tube-metal temperatures are quite even across the entire width of the superheater.

There is a great temptation on the part of many operators to try to eliminate recirculation at low loads. At first glance it might appear to be satisfactory to operate the unit at some partial rating without flue-gas recirculation. However, firing the fuel in the furnace with no cooling or heat-absorbing surface results in high furnace temperatures even at partial rating. Even though the load were only a relatively small percentage of maximum load, resulting in a correspondingly small amount of flue gases passing over the tubes, the steam flow through the tubes likewise would be correspondingly low. In many cases this would result in metal temperatures at the front end of the superheater above safe operating limits. Generally speaking, it is not wise even at partial loads to operate a unit designed for recirculation without the recirculation feature functioning, unless very careful consideration is given to all the

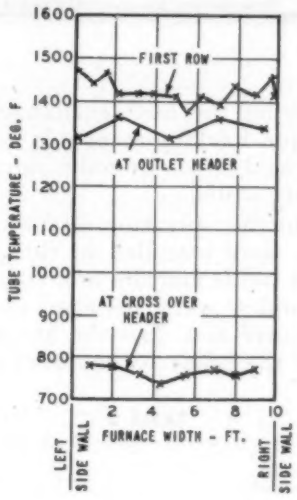

FIGURE 5

factors and possible pitfalls of a particular installation.

In conclusion, experience has shown that flue-gas recirculation is a practical and efficient application for the tempering of combustion gases before entering heat-exchange equipment, provided effective mixing of the gases is obtained by careful design. With the trend toward the use of increased temperatures for process and power purposes, its use should continue to increase steadily. Flue-gas recirculation has many applications. As described above it is excellently suited for separately-fired superheaters and air heaters. It is applicable also to special process equipment such as benzene superheaters in the plastic industry, and for oil heaters in the petroleum industry. In the chemical industry the temperature of chemical-bearing gases may be controlled by recirculation in the cooling-down process. In the power field where space conditions prevent the use of sufficient radiant-heat-absorbing surface to cool the gases below the fusion point of the ash, flue gas recirculation may be used to cool the gases and prevent slagging trouble. Finally flue-gas recirculation may play an important part in certain types of gas turbine applications.

Hydrocarbons In Petroleum

By FREDERICK D. ROSSINI

Thermochemistry and Hydrocarbons, Section Chief
National Bureau of Standards

FOR some years there has been under way in the petroleum industry a transition from the production of a few exceedingly broad fractions of petroleum, having general properties—such as the gas, gasoline, kerosine, lubricating oil, and wax—to the manufacture of many different narrow cuts having specialized properties. The general properties of each of the broad fractions have been such as to satisfy fairly well the requirements of a number of different services. However, the need for greater efficiency, higher speeds, and more concentrated power, the use of high pressures and high and low temperatures, and the invention and development of devices for improving the comfort and welfare of man, inevitably lead to the necessity of manufacturing materials of specialized properties which can better fill the ever-expanding and more exacting requirements of today.

Although investigations on the nature of petroleum have been going on for many decades, it was not until about 25 or 30 years ago that the petroleum industry itself began to consider seriously the chemical constitution of its raw material. With a few notable exceptions, the researches on the hydrocarbons in petroleum had been sporadic and casual; and relative to the importance, magnitude, and complexity of the problem, greatly limited in scope. About 25 years ago the presence of a number of hydrocarbons in the gasoline fraction of petroleum was reported, but for molecules of more than five carbon atoms the evidence was in most cases based upon the products obtained by chemical treatment of certain petroleum fractions or upon volume peaks in the distillation curve. What was needed was a comprehensive and unified investigation of the chemical constituents based upon the actual isolation of pure hydrocarbons from the crude oil.

In 1927 a comprehensive investigation on the isolation and identification of hydrocarbons in petroleum was begun at the National Bureau of Standards, sponsored jointly by the bureau and the American Petroleum Institute. The investigation has to date involved a total of about 200 man-years of research and has resulted in the separation of 72 hydrocarbons from petroleum. At the present time the staff of the American Petroleum Institute at the bureau consists of 10 research chemists and 12 laboratory assistants, supervised by the author. The investigations, under A. P. I. Research Project 6, are advised by a special committee appointed by the American Petroleum Institute.

All of the hydrocarbons in petroleum are composed of paraffin, cycloparaffin (naphthene), aromatic, or mixed cycloparaffin aromatic groups, or combinations of these. No olefin or acetylene hydrocarbons appear to occur naturally in petroleum to any significant extent. The problem is to separate the complex mixture of hydrocarbons which is petroleum and to obtain therefrom the unaltered major components in a substantially pure state.

THE accompanying report is a summary of the work of the American Petroleum Institute Research Project 6 at the National Bureau of Standards. The research staff engaged in this work consists of Frederick D. Rossini, Beveridge J. Mair, Charles B. Willingham, Augustus R. Glasgow, Jr., Alphonse F. Forziati, Anton J. Streiff, Morton B. Epstein, Vincent A. Sedlak, Evelyn T. Murphy, A. J. Sweetman, Janice C. Cahill, and Laurel F. Soule.

To be successful, any process of fractionation must involve a physical property that has significantly different values for the several components of the mixture to be separated. Furthermore, the process must be one for which there is available, or can be developed, a workable apparatus.

The standard processes of fractionation used in this work are distillation, extraction, adsorption, and crystallization, which depend, respectively, on the properties of vapor-pressure (or boiling point), solubility, adsorbability, and freezing point. The following are the variations which are used:

I. Distillation
1. At one fixed pressure
2. Alternately at two fixed pressures
3. With an azeotrope-forming substance

II. Extraction
1. With different solvents
2. At different temperatures

III. Adsorption
1. With different adsorbents
2. At different temperatures

IV. Crystallization
1. From the mixture of hydrocarbons, with centrifuging
2. From a solution of the hydrocarbons in an appropriate solvent, with centrifuging.

With respect to the nature of the separation attainable, the processes of fractionation (excluding crystallization) may be divided according to whether they fractnoiate primarily with respect to size of molecule or to type of molecule. Separation primarily according to size of molecule (or boiling point or molecular weight) is effected by distillation at one fixed pressure. Separation primarily according to type of molecule is effected by distillation alternately at two fixed pressures, by distillation with an azeotrope-forming substance, by extraction, and by adsorption. In connection with separation by crystallization, two general statements may be made regarding hydrocarbons: (a) for a given number of carbon atoms in the molecule, the freezing point usually increases with increase in the symmetry of the molecule; (b) for a given degree of symmetry of the molecule, the

freezing point usually increases with increase in the size of the molecule.

Except for those hydrocarbons of low molecular weight which in boiling point are relatively far removed from their neighbors, and except for certain other hydrocarbons which are present in relatively large amounts (as normal paraffins in a Pennsylvania or Mid-Continent petroleum), regular distillation alone is not sufficient to separate a pure hydrocarbon from petroleum, and one or more of the other methods of fractionation must also be used. The order in which the several methods are used is determined by the effectiveness of a given process in increasing the separation attained by the preceding method. Thus, while a given process may be ineffective when used alone, its systematic use in appropriate combination with other processes can often lead to the complete separation of an otherwise unseparable constituent.

Distillation is the most important fractionating process for separating hydrocarbons of the gasoline and kerosine fractions. This is because distillation can separate either by size or by type of molecule, according to whether one operates at a fixed pressure on the one hand, or alternately at two fixed pressures, or with an azeotrope-forming substance, on the other hand. For each of the several kinds of distillation the same rectifying columns may be used, with substantially the same procedure.

In the distillation laboratory at the National Bureau of Standards there are 15 rectifying columns in continuous operation, 24 hours per day, 7 days per week, every day in the year. These columns have separating efficiencies equivalent to from about 125 to 200 theoretical plates and are usually operated with reflux ratios from 125:1 up to 200:1, while the rate of removing distillate is from 2 to 12½ ml of liquid per hour. The time of distilling a given charge may vary from 600 to 2100 hours of continuous operation.

When a mixture of two hydrocarbons of different types but of the same normal boiling point is obtained by distillation at the normal pressure, it frequently can be resolved by alternately distilling at the normal pressure and at a reduced pressure. When the normal pressure is 760 mm Hg and the reduced pressure is 100 mm Hg, the downward displacement of the boiling point for several types of hydrocarbons, referred to the normal paraffins of the same normal boiling point as zero, is roughly as follows: normal paraffins 0° C.; isoparaffins, 0 to 2.5° C.; cycloparaffins (naphthenes), 1.5 to 4.5° C.; alkylbenzenes, 0 to 2° C.; alkylnaphthalenes, 3 to 4° C. Differences of 1° C. are sufficient to produce separation in a column of high efficiency.

Distillation with an azeotrope-forming substance is useful for separating hydrocarbons of about the same boiling point but of different types. Most polar organic molecules form minimum-boiling azeotropic mixtures with hydrocarbons. That is to say, in such mixtures, the given hydrocarbons have partial pressures greatly in excess of what they normally would have in an ideal solution at the same temperature and concentration. Comparing compounds of the same normal boiling point, the greatest excess of partial pressure is produced in the paraffins, next greatest in the cycloparaffins (naphthenes), with the monoolefins, diolefins, mononuclear aromatics, and dinuclear aromatics usually following in that order. For example, the binary azeotropic mixtures of ethanol (78° C.) with 2,4-dimethylpentane (80.8° C.), cyclohexane (80.8° C.), and benzene (80.1° C.) boil at about 62, 64 and 68° C., respectively. In general, aromatic hydrocarbons are easily separated from paraffins and cycloparaffins by azeotropic distillation, while the latter two are separated from each other with some difficulty.

The process of extraction is also used to separate hydrocarbons of about the same boiling point but of different types. As in the case of azeotropic distillation, aromatic hydrocarbons are easily separated from paraffins and cycloparaffins by efficient extraction, whereas paraffins and cycloparaffins are separated from each other with some difficulty. Three 56-foot glass columns of about 1 cm. diameter were used on aromatic-free material of the kerosine fraction to separate paraffins from naphthenes (more soluble) by selective extraction with reflux, with methyl formate as the solvent at a temperature near 10° C. The denser solvent phase descends through an ascending stream of fine globules of the hydrocarbon phase. For solvents lighter than the hydrocarbons being separated, the flows are reversed.

The process of adsorption is mainly used at present to separate aromatic hydrocarbons from paraffins and cycloparaffins, which separation can be made quantitatively. One of the great advantages of the process is that it can be operated successfully on wide-boiling charges of material, such as the entire gasoline fraction of petroleum. This process has been used effectively, in combination with the process of distillation, to analyze the hydrocarbons in the gasoline fraction of seven different representative petroleums.

The process of crystallization generally is used to increase the purity of a hydrocarbon which has been concentrated by one or more of the other fractionating processes. The more frequently used procedure is to mix the hydrocarbon with the proper amount of a suitable solvent, take the mixture (usually by refrigeration) to a temperature that produces a "mush" or "slurry" of liquid with crystals of the wanted hydrocarbon, and then at this temperature to centrifuge the liquid from the crystals. In some cases, especially when it is sufficiently fluid near its melting point, the liquid hydrocarbon may be processed without solvent, the crystals being freed from the mother liquor by centrifuging as before.

After a given hydrocarbon has been separated from petroleum in the manner just described, a number of physical properties are measured for record and identification. These properties include the boiling point, freezing point, density, refractive index, and refractive dispersion. When the identity of the hydrocarbon is not known, determinations of the molecular weight and the carbonhydrogen content are also made. Other properties which are sometimes determined include the critical solution temperature in aniline, the viscosity, and the infrared absorption spectrum.

Any rational method of purification will leave in a hydrocarbon only those impurities that have properties close to those of the given hydrocarbon, and such impurities will tend to form an ideal solution with it. Furthermore, such impurities seldom form solid solutions with the given hydrocarbon. The ideal solution laws may, therefore, be applied to freezing and melting observations on such systems to ascertain quantitatively the amount of the unknown impurity. No property other than the freezing point can be so translated quantitatively into amount of impurity when the impurity is unknown.

The amount of impurity in the purest lot of the

TABLE 1

Hydrocarbons separated from a Mid-Continent Petroleum by the A.P.I. Research Project 6 at the National Bureau of Standards

No.	Formula	Name	Type	B.P. °C.
1	CH_4	Methane	Paraffin	—161.6
2	C_2H_6	Ethane	Paraffin	— 88.9
3	C_3H_8	Propane	Paraffin	— 42.1
4	C_4H_{10}	isobutane	Paraffin	— 11.7
5	C_4H_{10}	n-butane	Paraffin	— 0.5
6	C_5H_{12}	2-methylbutane	Paraffin	27.8
7	C_5H_{12}	n-pentane	Paraffin	36.1
8	C_5H_{10}	Cyclopentane	Cycloparaffin	49.5
9	C_6H_{14}	2,3-dimethylbutane	Paraffin	58.0
10	C_6H_{14}	2-methylpentane	Paraffin	60.3
11	C_6H_{14}	3-methylpentane	Paraffin	63.3
12	C_6H_{14}	n-hexane	Paraffin	68.7
13	C_6H_{12}	Methylcyclopentane	Cycloparaffin	71.9
14	C_7H_{16}	2,2-dimethylpentane	Paraffin	79.2
15	C_6H_6	Benzene	Aromatic	80.1
16	C_6H_{12}	Cyclohexane	Cycloparaffin	80.8
17	C_7H_{14}	1,1-dimethylcyclopentane	Cycloparaffin	87.5
18	C_7H_{16}	2-methylhexane	Paraffin	90.0
19	C_7H_{14}	Trans-1,3-dimethylcyclopentane	Cycloparaffin	90.9
20	C_7H_{14}	Trans-1,2-dimetnylcyclopentane	Cycloparaffin	91.9
21	C_7H_{16}	3-methylhexane	Paraffin	92.0
22	C_7H_{16}	n-heptane	Paraffin	98.4
23	C_7H_{14}	Methylcyclohexane	Cycloparaffin	100.9
24	C_7H_{14}	Ethylcyclopentane	Cycloparaffin	103.5
25	C_8H_{16}	1,1,3-trimethylcyclopentane	Cycloparaffin	105.0
26	C_8H_{18}	2,2-dimethylhexane	Paraffin	106.8
27	C_7H_8	Toluene	Aromatic	110.6
28	C_8H_{18}	2-methylheptane	Paraffin	117.6
29	C_8H_{16}	1,1-dimethylcyclohexane*	Cycloparaffin	119.8
30	C_8H_{16}	Trans-1,3-dimethylcyclohexane	Cycloparaffin	120.3
31	C_8H_{16}	Trans-1,2-dimethylcyclohexane*	Cycloparaffin	123.4
32	C_8H_{18}	n-octane	Paraffin	125.6
33	C_8H_{16}	Ethylcyclohexane	Cycloparaffin	131.8
34	C_9H_{20}	2,6-dimethylheptane	Paraffin	135.2
35	C_8H_{10}	Ethylbenzene	Aromatic	136.2
36	C_9H_{18}	1,1,3-trimethylcyclohexane	Cycloparaffin	136.7
37	C_8H_{10}	p-xylene	Armcatic	138.4
38	C_8H_{10}	m-xylene	Aromatic	139.2
39	C_9H_{20}	2,3-dimethylheptane*	Paraffin	140.7
40	C_9H_{18}	1,2,4-trimethylcyclohexane	Cycloparaffin	141.2
41	C_9H_{20}	4-methyloctane	Paraffin	142.4
42	C_9H_{20}	2-methyloctane	Paraffin	143.3
43	C_9H_{20}	3-methyloctane	Paraffin	144.2
44	C_8H_{10}	o-xylene	Aromatic	144.4
45	C_9H_{18}	Menocyclic†	Cycloparaffin	145.6
46	C_9H_{16}	Bicyclic†	Cycloparaffin	146.7
47	C_9H_{20}	n-nonane	Paraffin	150.7
48	C_9H_{12}	iso-propylbenzene	Aromatic	152.4
49	C_9H_{12}	n-propylbenzene	Aromatic	159.5
50	C_9H_{12}	1-methyl-3-ethylbenzene	Aromatic	161.6
51	C_9H_{12}	1-methyl-4-ethylbenzene	Aromatic	162.5
52	C_9H_{12}	1,3,5-trimethylbenzene	Aromatic	164.6
53	C_9H_{12}	1,methyl-2-ethylbenzene	Aromatic	165.1
54	$C_{10}H_{22}$	4-methylnonane	Paraffin	165.7
55	$C_{10}H_{22}$	2-methylnonane	Paraffin	166.8
56	$C_{10}H_{22}$	3-methylnonane	Paraffin	167.8
57	C_9H_{12}	1,2,4-trimethylbenzene	Aromatic	169.2
58	$C_{10}H_{22}$	n-decane	Paraffin	174.0
59	C_9H_{12}	1,2,3-trimethylbenzene	Aromatic	176.1
60	$C_{11}H_{16}$	n-amylbenzene*	Aromatic	204.1
61	$C_{10}H_{14}$	1,2,3,4-trimethylbenzene	Aromatic	205.0
62	$C_{11}H_{16}$	1,4-dimethyl-2-propylbenzene*	Aromatic	206.6
63	$C_{10}H_{12}$	5,6,7,8-tetrahydronaphthalene	Aromatic-cycloparaffin	207.6
64	$C_{11}H_{16}$	1,5-dimethyl-2-propylbenzene*	Aromatic	208.5
65	$C_{11}H_{16}$	1,3,5-trimethyl-2-propylbenzene*	Aromatic	212.3
66	$C_{12}H_{26}$	n-dodecane	Paraffin	216.3
67	$C_{10}H_8$	Naphthalene	Aromatic	218.0
68	$C_{12}H_{16}$	Phenylcyclopentane*	Aromatic-cycloparaffin	220.7
69	$C_{11}H_{14}$	2-methyl-5,6,7,8-tetrahydronaphthalene	Aromatic-cycloparaffin	229.0
70	$C_{11}H_{14}$	1-methyl-5,6,7,8-tertahydronaphthalene	Aromatic-cycloparaffin	234.4
71	$C_{11}H_{10}$	2-methylnaphthalene	Aromatic	241.1
72	$C_{11}H_{10}$	1-methylnaphthalene	Aromatic	244.8

† Identity not yet determined. * Identity not definitely established.

hydrocarbon is determined by means of time-temperature freezing and melting curves. A large number of the hydrocarbons isolated in this work have contained 0.01 or less mole fraction of impurity and some contained 0.001 or less mole fraction.

In Table 1 are listed the 72 hydrocarbons which have so far been separated from the gas, gasoline and kerosine fractions of a Mid-Continent petroleum by API Research Project 6.

In addition to the actual isolation of pure hydrocarbons from the kerosine and more volatile fractions, considerable work has been done on the lubricant fraction of the same petroleum to produce fractions of material substantially homogeneous with respect to size and type of molecules.

In this work, the lubricant fraction was separated by distillation, crystallization, extraction, and adsorption into 4 broad portions: a "wax" portion, 35 percent, which was pure hydrocarbon material and contained a considerable amount of clear oil in addition to the true wax; a "water-white" oil portion, 35 percent, which was pure hydrocarbon material; an "extract" portion, 22 percent, which contained, by weight, about 0.9 percent S, 0.1 percent N; and 0.5 percent O; "asphaltic" portion, 8 percent, which contained the bulk of the nonhydrocarbon components and was mostly a black solid at room temperature. The "wax" and "asphaltic" portions were not further separated, but the "water-white" oil and "extract" portions were each subjected to an exhaustive separation with respect to both size and type of molecules. A large number of final fractions substantially homogeneous with respect to size and type of molecules were obtained in this work. These final fractions were about 15 grams in size and each represented 1/40,000th part of the original crude petroleum from which it came.

As a result of correlating the properties of these final fractions with those of known hydrocarbons, the following average analysis was made with regard to the kinds of molecules in the middle of the original lubricant fraction: normal paraffins (plus possibly a small amount of isoparaffins), 18 to 26 percent, naphthenes or cycloparaffin hydrocarbons with 1, 2, or 3 rings per molecule, 51 to 43 percent; mixed cycloparaffin-aromatic hydrocarbons with 1 aromatic ring and, in the same molecule, 1, 2, or 3 cycloparaffin rings, 8.3 percent; mixed cycloparaffin-aromatic hydrocarbons, with 2 aromatic rings (condensed as in naphthalene) and, in the same molecule, 2 cycloparaffin rings, 8.1 percent; mixed cycloparaffin-aromatic hydrocarbons with 3 aromatic rings (condensed) and, in the same molecule, 1 cycloparaffin ring, 6.6 percent; "asphaltic" constituents, 8 percent. The striking part of this analysis of the lubricant fraction is the absence of any significant amount of purely isoparaffin hydrocarbons, and, except in the "asphaltic" portion, of any purely aromatic hydrocarbons.

The researches necessary to carry on a petroleum chemical industry whose purpose is substantially that of achieving a more economic utilization of petroleum, may logically be divided into two groups: fundamental research and applied researches. The fundamental researches include the investigation of the chemical constitution of petroleum and the determination of the physical and thermodynamic properties of hydrocarbons and their derivatives, and the study of the mechanisms of their reactions. The applied researches include the development of practical commercial methods for effecting the separation of wanted hydrocarbons and their derivatives from petroleum and petroleum products, and the investigation of various chemical reactions involving petroleum hydrocarbons and their derivatives for the purpose of obtaining new and desirable substances. The fundamental researches should be carried on and encouraged in the university and government laboratories, leaving the competitive research and development units of the industry to make practical application of the knowledge and data obtained by the non-profit laboratories. The system should be a self-perpetuating one wherein the industry, profiting from its applications of the basic knowledge and data, would be anxious to assure the continued flow into the proper channels of resources for researches on base-line problems.

The Rotameter and Petroleum Refinery Flow Rate Measurement

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UP TO the present time the potential advantages and improvements which the variable-area flow-rate meter (rotameter) offers the petroleum refinery engineer for increasing the accuracy, simplicity and, above all, the scope of process control have gone almost totally unrecognized. True, the typical refinery engineer is aware of the fact that "a lot of rotameters have been sold in the last 10 years." Moreover, he has a few rotameters, usually simple direct-indicating ones, on some of his chemical-feed lines. However, that many of the now most baffling control problems and even some which no attempt is being made to solve, can be mastered easily by means of rotameters has unfortunately not been demonstrated to him due to the preoccupation of rotameter manufacturers with filling the evergrowing needs of the chemical process industries and, from 1941 to 1945, with special war-effort problems.

The fact that the chemical industries have absorbed almost the entire output of rotameters ever since the writer first began building them by hand back in 1931 is accounted for by three obvious considerations:

1. Rotameters combine the function of the sight glasses frequently needed in process lines with the function of a flow-rate meter, thus saving a piece of equipment.

2. Complete corrosion resistance can be obtained easily since the rotameter metering tube usually is made of borosilicate glass.

3. Impairment of proper operation by dirt and solids can be visually noted without breaking the piping system or shutting down.

Sufficient benefit was derived from these elementary advantages that a number of other advantages of a more fundamental nature, though only slightly less obvious in character, were overlooked for many years. This occurred in spite of the fact that these advantageous factors as listed below are so distinctly lacking in variable-head-type flow-rate meters (orifice, nozzle and venturi).

4. No limitations on the piping layout are imposed since no straight runs of pipe or straightening vanes are required adjacent to rotameters. Valves or elbows may be placed as near the instruments as desired without impairing calibration accuracy.

5. The calibration plot of a rotameter is practically linear instead of being quadratic as in the case of variable-head meters. Thus capacity-scale graduations are uniformly spaced and are read as accurately at low flows as at high flows.

6. No manometer and no manometer taps are needed with rotameters. The entire unit fits right into the piping system. Thus the whole problem of carefully drilling and reaming tap connections, providing seal pots, condensate pots and valves, mounting the manometer, and keeping all these parts free of foreign matter is eliminated.

7. No mercury (or any other accessory "metering fluid") is required to operate rotameters. Thus corrosion problems are simplified and the problem of safeguarding and retaining the mercury disappears.

8. The pressure loss across any one rotameter remains constant regardless of fluid-flow rate, after the float dimensions and weight have been selected. It does not increase with the square of the flow rate as in orifice meters. Furthermore, the pressure-loss value may be selected as small as desired—even down to less than one-inch water column.

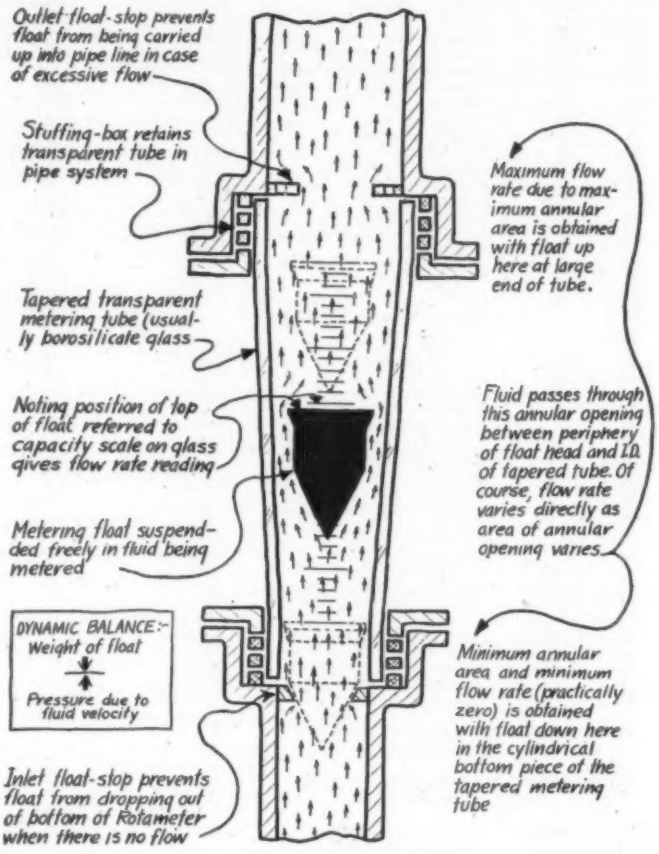

Outlet float-stop prevents float from being carried up into pipe line in case of excessive flow
Stuffing-box retains transparent tube in pipe system
Tapered transparent metering tube (usually borosilicate glass
Noting position of top of float referred to capacity scale on glass gives flow rate reading
Metering float suspended freely in fluid being metered
DYNAMIC BALANCE:- Weight of float
Pressure due to fluid velocity
Inlet float-stop prevents float from dropping out of bottom of Rotameter when there is no flow
Maximum flow rate due to maximum annular area is obtained with float up here at large end of tube.
Fluid passes through this annular opening between periphery of float head and I.D. of tapered tube. Of course, flow rate varies directly as area of annular opening varies.
Minimum annular area and minimum flow rate (practically zero) is obtained with float down here in the cylindrical bottom piece of the tapered metering tube

FIGURE 1
The principle of the rotameter

9. A flow range as great as 20:1 can be handled in a single rotameter. That is, a minimum flow rate as low as one twentieth of the maximum can be measured without impairment of accuracy.

10. The smallest flow rates, down to fractions of a milliliter per minute of liquid or gas, are easily handled with rotameters.

Rotameters embodying these advantageous characteristics have been used in oil refineries for controlling the injection of ammonia into crude-oil lines

to obtain corrosion control, for metering caustic soda and sulphuric acid in gasoline-treating processes, for setting reflux-product ratio, side draw-off rate and feed rate in light-oil distillation units, for metering chlorine for algae control in cooling-water systems, for metering catalyst slurries, hydrofluoric acid, acid sludges, and many other liquids as well as vapors and gases.

The Valve Body "Rotaline" Meter

During the last year, however, a number of improvements have been devised to make the rotameter fully suitable for the heavy-duty requirements of the petroleum refineries. Notable among these is the rugged "Rotaline" instrument which is built like a globe valve, having flanged horizontal inlet and outlet connections. The metering tube is accessible through cover flanges directly above and below it. To these access flanges sight extensions and remote transmitters may be attached. If a glass metering tube is used it is supported in a metal lantern with which it withdraws as a unit in case it is to be removed from the meter body. The lantern is arranged so that the fluid being metered is in contact with both the inside and outside of the metering tube and no differential fluid pressure, therefore, has to be withstood by the glass. Metal metering tubes may also be used interchangeably with the lantern-glass tube assemblies. Two plugged alignment holes are provided through which the zero calibration mark on the metering tube may be observed and the proper dimensional spacing of tube, float, sight extension and remote transmitter thus assured. By means of an adjusting nut at the bottom of the lantern the metering tube position may be vertically altered. The extension rods attached to the metering floats for observation in the sight windows or for actuating remote transmitter are only half as long as in older types of armored rotameters. Thus there is no longer any trouble trying to "fish" long float assemblies into the meter bodies, no danger of damaging the float edges or of bending and kinking the extensions and thus causing friction. The compactness of the meter body is commensurate with that of the float assemblies, being approximately one half that of older meters. Weight reduction is at least one third, but any refinery pressure or temperature can be handled since the weight reduction is accomplished by compact design, not by skimping.

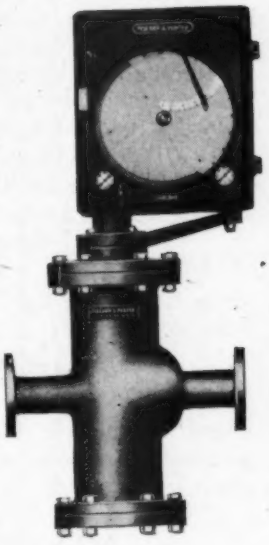


FIGURE 2
A valve-body "Rotaline" with close-coupled Recorder, Integrator and Flow Controller.

The Bead-Guide Tube

Another new mechanical improvement has thoroughly solved the problem of float guiding. As floats which are dynamically stable and do not require guides have a number of disadvantages (which will be discussed later) it was formerly necessary to use metalic axial guide wires which were run through the floats. These had to be located very precisely on the length-wise axis of the tubes to prevent the floats from striking the glass when near the small end of the tubes and this necessitated the use of centering spiders. The wires also had to be kept taut, which requirement brought with it the need for tightening nuts, locknuts and threading of the wires. A mirror-smooth hard surface, to minimize friction, was needed on the guide wires and the bushings which were inserted in the floats. Yet the guide wire assemblies had to be made of the most difficultly machined metals in consideration of corrosive fluids to be handled. The bead-guide tube eliminates a minimum of five metal parts, provides a guiding surface harder than any metal, gives a coefficient of friction less than a third that obtainable with polished metal. There is no longer any possibility of misalignment trouble and the floats can not possibly strike sharply against the glass tubes since the clearance between their edges and the inner surfaces of the beads is only about 0.002 inch.

FIGURE 3
A typical "Bead-Guide" Rotameter Tube.

Opaque Fluid Measurement

The bead-guide tube also solves the problem of retaining complete float visibility when metering the opaque fluids frequently encountered in refinery work. Since the film of liquid between the bead edge and the reading edge of the float is so thin even the darkest oil will be at least translucent in this area. Formerly sight extensions had to be built onto rotameters when they were to be used for opaque-fluid measurement. By eliminating this extra attachment the bead-guide tube sharply reduces the cost of opaque fluid rotameters.

Viscosity Immunity

Since the summer of 1940 research work has been steadily carried on for the purpose of making rotameter readings independent of variations in viscosity of the fluids metered. Although not considered completed, this work has put the rotameter so very far ahead of the best variable-head-type meter as to beggar comparison. Whereas a Reynolds number of 2000 must always be exceeded and 10,000 must usually be exceeded to insure constant flow coefficients for orifice-type meters it is no problem to obtain constant coefficients with rotameters at Reynolds numbers as low as 400. In some cases it has even been possible to go below 100 Reynolds number before noting a coefficient change. Since the Reynolds number varies inversely with viscosity (Reynolds number = density $\times$ velocity $\times$ pipe diameter/viscosity) it will be apparent that the Rotameter will operate without reading error with viscosity changes at least five times as great, and frequently ten or twenty times as great, as will cause a calibration change in an orifice meter. In oil refineries, where there is not only a substantial difference between viscosities of different fluids, but where the viscosity of one fluid will vary considerably from hour to hour, the high degree of viscosity immunity obtainable with rotameters is particularly important. In larger lines the viscosity immunity of rotameters is higher—where viscosity is measured in S.S.U. or centipoise—because the Reynolds numbers will be higher. Immun-

ity all the way from 30 S.S.U. to 1500 S.S.U. has been obtained in 4-inch lines. However, even with flows as low as 1 gallon per minute in ½-inch lines good immunity is still obtainable.

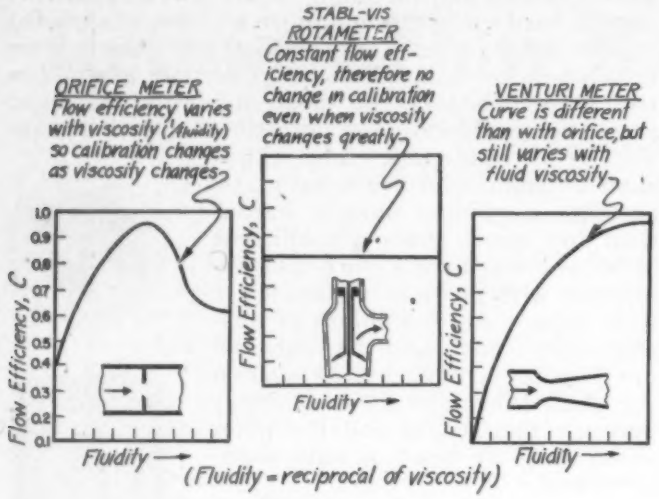

ORIFICE METER
Flow efficiency varies with viscosity (Fluidity) so calibration changes as viscosity changes
STABL-VIS ROTAMETER
Constant flow efficiency, therefore no change in calibration even when viscosity changes greatly
VENTURI METER
Curve is different than with orifice, but still varies with fluid viscosity
Flow Efficiency, C
Fluidity
(Fluidity = reciprocal of viscosity)

FIGURE 4

Graph to show viscosity immunity (constancy of flow coefficient with varying Reynolds Number) of rotameter compared to orifice, nozzle and venturi meters.

Continuous Viscosimeters

While discussing viscosity immunity it seems appropriate to digress for a moment from a discussion of flow-rate measurement to take up the latest use of rotameters in petroleum refineries: The continuous measurement of viscosity of fluids flowing in piping systems. To accomplish this an otherwise standard rotameter is equipped with two floats. The lower one is made to have the highest degree of viscosity immunity while the upper one is made very viscosity sensitive. Thus a given flow rate will always lift the upper float a greater distance than the lower one because the upper one is acted upon both by velocity head and viscous drag, whereas the lower one is lifted only by velocity head. Thus the distance between the two floats is a measure of viscosity. If the lower float is held at some predetermined index setting corresponding to one particular flow rate the tube scale may be graduated so that the position of the upper float corresponds directly to viscosity readings. Rotameters for continuous viscosity measurement may be inserted directly in the main flow lines if these are reasonably small.

FIGURE 5

Diagram of a Rotameter Type Automatic Continuous Viscosimeter.

For larger pipe sizes the rotameter may be inserted in a bypass. The adjusting of the flow rate to set the viscosity-immune float at its index height may be done manually when it is desired to take a viscosity reading or an automatic flow controller may be placed in the bypass line to maintain a constant small flow through the rotameter-viscosimeter regardless of flow variations through the main line. Automatic controls also may be incorporated to maintain viscosity constant by altering the flow of heating steam or blending fluid. Whatever the arrangements selected may be, it is unnecessary to withdraw fluid samples from the piping system or to take intermittant and repeated efflux time runs in some laboratory-type apparatus. Thus one of the most annoying vacancies in the roster of available industrial instruments is filled.

Automatic "Inherent" Density Compensation

Returning now to strict flow-rate measurement let us take up one of the most unusual and tantalizing characteristics of the rotameter: Its ability to measure flow rate of fluids of varying density without change in calibration as the density varies. This characteristic could formerly be built into rotameters only for the measurement of liquids in gravimetric units. Recently, however, it has also been made available in meters handling gases and vapors (including steam). Even more recently a method has been devised for imparting "inherent" density compensation to rotameters measuring liquids in volumetric units. To understand how "inherent" density compensation is obtained it is necessary to understand clearly the mechanism of the rotameter. The metering float is in dynamic equilibrium, the downward force, equal to effective float weight, or float volume × float density minus fluid density, is in dynamic balance with the upward force, pressure drop across float × area of flow at largest periphery, or:

$$V_f(\gamma_f - \gamma_w) = (P_1 - P_2)\,A_f \quad \ldots\ldots\ldots\ldots 1$$

since $h = v^2/2g$ by Torricelli's equation, we write for substitute and transpose to obtain

$$\frac{V_f(\gamma_f - \gamma_w)}{A_f \gamma_w} = h \quad \ldots\ldots\ldots\ldots 2$$

since $h = v^2/2g$ by Torricelli's equation we write for fluid velocity:

$$V = \left[\frac{2g\,V_f(\gamma_f - \gamma_w)}{A_f \gamma_w}\right]^{1/2} \quad \ldots\ldots\ldots\ldots 3$$

since volumetric flow rate is velocity times area available for flow we obtain, when efficiency coefficient C is added

$$Q = A_w C\left[\frac{2g\,V_f(\gamma_f - \gamma_w)}{A_f \gamma_w}\right]^{1/2} \quad \ldots\ldots\ldots\ldots 4$$

Now to obtain gravimetric (weight) rate of flow we must multiply by fluid density to obtain

$$Q\gamma_w = W = A_w C\left[\frac{2g\,V_f(\gamma_f - \gamma_w)(\gamma_w)}{A_f}\right]^{1/2} \quad \ldots\ldots\ldots\ldots 5$$

Obviously the effect of changing float or fluid density is contained in the expression $[(\gamma_f - \gamma_w)(\gamma_w)]^{1/2}$ and we note that fluid density γ_w is contained in this expression twice, once as γ_w and once as $(\gamma_f - \gamma_w)$. As the fluid density increases γ_w increases but $(\gamma_f - \gamma_w)$ decreases. This brings up the possibility of selecting values for γ_w such that the changes in the two factors will balance each other out. If we construct a graph on which fluid density γ_w as abscissa is plotted against change in calibration due to density change $[(\gamma_f - \gamma_w)(\gamma_w)]^{1/2}$ as ordinate then the slope of the curve obtained is the derivative of $[(\gamma_f - \gamma_w)(\gamma_w)]^{1/2}$ respective to γ_w. But we also know that a curve is flat and horizontal where the derivative equals zero and we find that to obtain this condition we must insert values for γ_f which are twice γ_w. Therefore: If we make the float density twice the fluid density no calibration change will be caused even by wide fluid-density changes. It is not really necessary to resort to calculus to understand this. Just insert a value of 2.0 for γ_f and then vary γ_w upward and downward from 1.0 to see how nearly constant the value of $[(\gamma_f - \gamma_w)(\gamma_w)]^{1/2}$ will remain.

When metering liquids the required 2:1 ratio is easily obtained by using floats made partly of metal and partly of plastics. Innumerable engine-test laboratories and production-test stands for gasoline engines, diesel engines and jet-propulsion engines use "inherently" compensated rotameters for instantaneously determining pounds of fuel used per hour without recourse to weighing tanks, stop watches or lengthy time runs. A number of these laboratories are located in refineries or are owned by petroleum refining companies.

When metering gases or vapors it is not quite so simple to obtain "inherent" density compensation. The density of stainless steel, for example, is some 13,000 times greater than the density of steam at atmospheric pressure. This problem is solved by constructing a variable-area meter as per Figure 6. To the lower end of the metering float is attached a rod on which is hung a hollow cylinder. The cylinder is located in a well into which steam condensate drains. There is buoys up the hollow cylinder and thus reduces the effective weight of the float. Since mercury has a density about 13.5 times that of water the size of the extension well and the hollow cylinder can be greatly reduced by filling the well with mercury. This has the further advantage of offering a means for correcting the errors which otherwise would occur if the temperature in the extension well changed sufficiently to appreciably change the density of the buoying liquid. Since the density of solid metal will change only very slightly with temperature changes, whereas mercury will change more and water still more, it is possible to add a skirt to the bottom of the buoying extension and insert enough water in it to make the expansion coefficient of the metal float and its water equal the expansion coefficient of the mercury. Figure 7 plots the calibration change for a typical density-compensated steam-flow meter as pressure changes from three mean values of 15, 100 and 300 psig pressure respectively. It will be seen by an inspection of this graph that the pressure regulation would have to be amazingly poor indeed to produce a significant calibration variation since a steam density variation of as much as ± 10 percent from a mean value does not produce a variation of more than ± 0.2 percent.

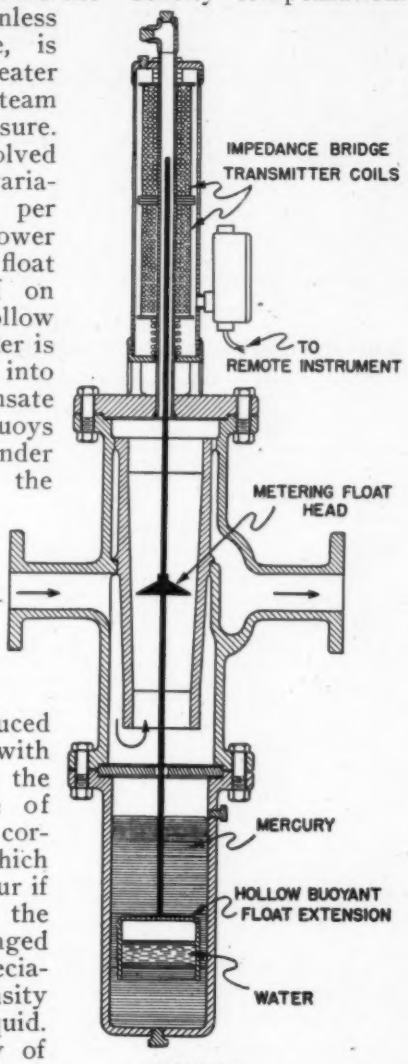


FIGURE 6
Sectional diagram of a Steam Rotameter with "Inherent" Automatic Compensation for Pressure and Temperature Changes and with Electric Remote Transmitter.

It is also to be noted that the variable-area type meter when used with widely varying temperature eliminates the problem of dimensional change of the metering aperture. Whereas in the variable-head meter growth of the orifice plate or nozzle will diminish

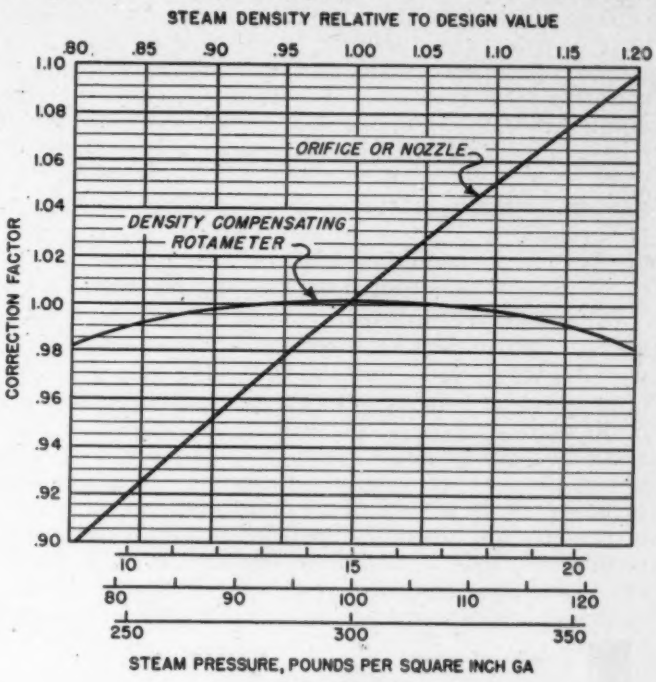

STEAM DENSITY RELATIVE TO DESIGN VALUE
.80 .85 .90 .95 1.00 1.05 1.10 1.15 1.20
1.10 1.08 1.06 1.04 1.02 1.00 .98 .96 .94 .92 .90
CORRECTION FACTOR
ORIFICE OR NOZZLE
DENSITY COMPENSATING ROTAMETER
10 15 20
80 90 100 110 120
250 300 350
STEAM PRESSURE, POUNDS PER SQUARE INCH GA

FIGURE 7
Effect of Steam Pressure and Density Changes on Calibration of Rotameter with "Inherent" Compensation.

the size of the flow aperture, growth of the float head in the variable-area meter will be accompanied by corresponding growth of the metering tube which should, of course, be made of the same material as the float.

Now let us return to the problem of obtaining "inherent" density compensation when metering liquids in volumetric units. From the equation for volumetric flow it is apparent that we must either give γ_f a value close to infinity or reduce γ_w to a value close to zero. Since the latter is impossible and the former impractical it would appear, at first sight, that "inherent" density compensation when metering liquids in volumetric units is out of the question. Suppose, however, that we try turning the rotameter up side down so that the fluid enters at the top and flows downward from the small to the large end of the metering tube. For operation in this position it becomes necessary that the metering float be less dense, rather than more dense, than the fluid, so that it will tend to rise to the small end of the tube. This will alter the factors in our equation dealing with float and float density to read $\left(\frac{\gamma_w - \gamma_f}{\gamma_w}\right)^{1/2}$ and it now is seen that float density must be reduced to zero if there is to be no effect on calibration of fluid density change. Since we can readily make a buoyant float and since it is much easier to approach zero float density than to approach infinity (as was shown to be necessary in an upright rotameter) we can attain a good degree of "inherent" density compensation. Taking for example a float specific gravity of 0.5 and a fluid specific gravity of 1.0 we find that a fluid specific gravity variation of ± 10 percent gives a calibration change of 4.5 percent while a variation

of — 10 percent gives a calibration change of 5.5 percent. If we reduce the float specific gravity to 0.3 the calibration change figures are reduced to 1.9 percent and 2.45 percent with + 10 percent and — 10 percent fluid specific gravity changes respectively. If we further reduce the float specific gravity to 0.25 the corresponding calibration changes reduce to 1.3 percent and 1.85 percent. Thus for a total fluid specific gravity change of ± 10 percent a calibration error of not over 1.85 percent from a mean value will be encountered. Of course, an inverted rotameter to give volumetric "inherent" density compensation when measuring liquids will be quite large due to the relatively low velocity past the float. However, if the gain in accuracy warrants it, the job can be accomplished.

Remote Instruments— Explosion-Proof Transmitting Systems

Rotameter readings have for many years been transmitted to remote instruments by means of the well-known electric impedance bridge systems. One outstanding advantage of remote-reading rotameters is the ability to check the reading of the primary and secondary instruments visually and at any flow rate, not merely at zero flow position. During the last three years, however, due to the justified antipathy of refinery engineers for instruments embodying explosion hazards, a magnetic transmitter was developed, and first installed in a West Texas petroleum refinery, which requires no electric supply whatsoever for operation and which is unique in so far as the two halves of the magnetic coupling cannot "loose" each other. Referring to Figure 8 it will be noted that a set of magnets is imbedded in a tube attached to the float. This tube moves in an outer tube closed at one end and communicating directly at the other end with the fluid-filled space in the rotameter (no stuffing boxes, pressure-tight bearings,

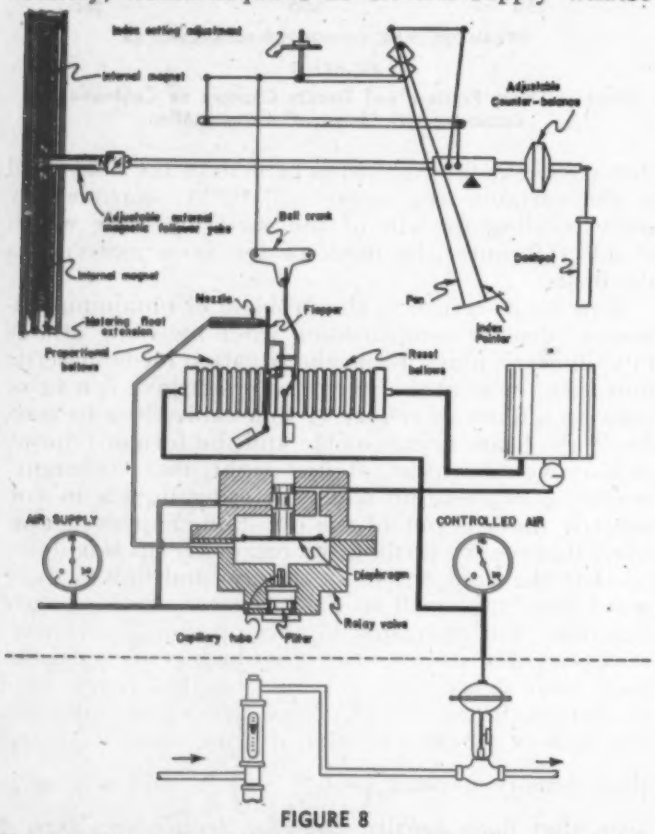


FIGURE 8

Magnetic "Magna-Bond" Transmitter for operating secondary instruments from Rotameter without use of electricity or of stuffing boxes.

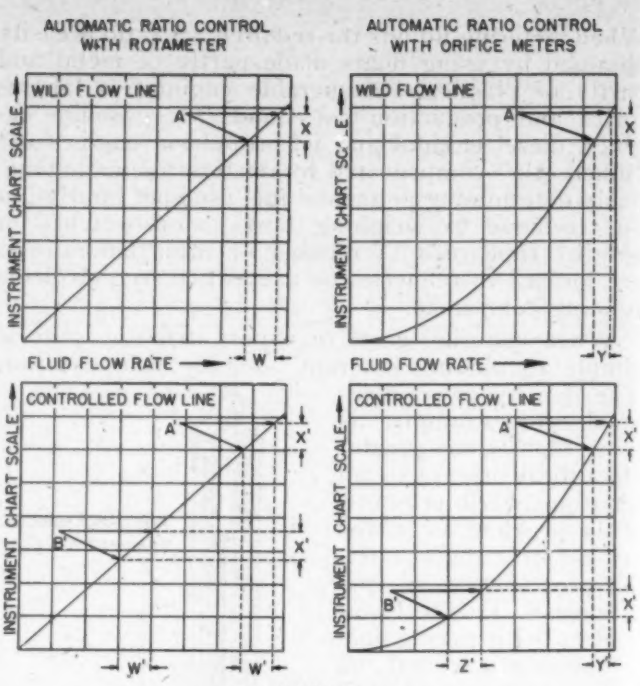


FIGURE 9

Effect of varying flow ratio on Variable Head Meters and on Rotameters.

The two graphs above give a comparison of automatic flow ratio control with variable head type (orifice type) flow meters and with variable area type (rotameter) flow meters. Referring to the left upper diagram, "A" represents the pen in the uncontrolled ("wild") flow line and "X" represents any given pen movement caused by the flow rate change "Y." Referring now to the lower left hand graph if the pen "A^1" is in the same relative position on the controlled flow line meter scale as pen "A" is for the wild line meter scale then the movement "X^1" of pen "A^1" (identical to pen movement "X" for pen "A") will result in a flow rate change "Y^1" exactly proportional to flow rate change "Y." However, if the pen showing controlled line flow is at some position "B^1" not corresponding on its meter scale to the position "A" of the pen showing the wild line flow then the pen movement "X^1," still exactly equal to pen movement "X" will result in a flow rate change "Z^1" having a magnitude much different than the flow rate change "Y" in the wild flow line. This illustrates the difficulty of coordinating orifice type flow rate meters for automatic ratio control unless the two flow rates are always maintained at exactly corresponding positions on the scales of the two flow-rate meters. Referring now to the two diagrams at the right it will be observed that with any given pen position "A" in the wild line of a rotameter automatic ratio control unit and with a given pen movement "X" produced by a flow rate change "W," the pen of the controlled line rotameter may be either at a position "A^1" corresponding to pen position "A" or at some entirely different position such as "B^1" and produce flow rate changes "W^1" having values proportional to the flow rate change "W" in the wild line provided, of course, that the pen movement is always "X^1" corresponding to the assumed pen movement "X" in the wild flow line. This result is obtained because of the linear calibration scales of the rotameters. The effect of operating with linear calibration scales with rotameters and square-root calibration scales with orifice-type flow-rate meters is clearly seen by comparing the diagrams at the right with those at the left.

etc.). These two magnets are arranged with their north poles adjacent to each other and the two south poles at the far ends of the assembly. The follower magnet, outside the fluid-filled tube, is placed crosswise respective to the leader magnets with its south pole nearer to the tube. The follower south pole is, of course, attracted to the two adjacent leader north poles and assumes a position corresponding to their junction. The nearer to the point of north pole junction the follower south pole comes, the greater is the attractive force. The further the follower south pole moves away from the junction the more does it encounter repulsion from the south poles of the leader magnetc. Thus it always is driven toward the junction point at the middle of the leader assembly and cannot "loose" the leader magnets even if a violent flow surge momentarily impairs correspondence. Close-coupled indicators, recorders and integrators are actuated directly from the follower magnet movement through suitable gearing. If remote instruments are required, pneumatic transmitters of conventional type are used.

Automatic flow controllers of conventional type are

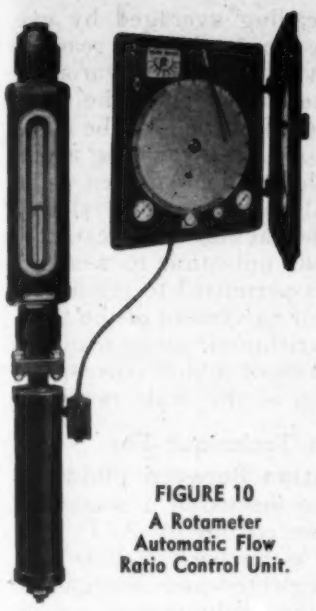


FIGURE 10
A Rotameter Automatic Flow Ratio Control Unit.

also available for use with rotameters. It is somewhat easier to obtain accurate flow control with rotameters than with variable-head flow-rate meters because of the rotameter's linear calibration scale. A given pen-movement increment represents the same deviation of flow rate from the control point at all capacities within the range of the instrument. While this is of minor importance in simple control of one fluid, it becomes of major importance when two or more fluids must be controlled in ratio with each other. When ratio control is attempted with variable-head flow meters, difficulty is to be expected as soon as the ratio is varied from the one predetermined figure which enables the "wild" and the "controlled" instruments to always operate at corresponding points (and corresponding slopes) on their calibration curves. However, if the ratio is varied, then equal increments of pen movement will no longer represent equal percentage increments of flow change. Thus the ratio maintained will vary as the flow rate in the wild line varies.

If we get too far down into the flat portion of the variable-head meter square-root calibration curve hunting and cycling will probably commence. It is undoubtedly for this particular reason that so little automatic ratio control has been used until the advent of the rotameter ratio controller and that variable-head-meter manufacturers, when attempting ratio control, have avoided direct coupling of the two instruments. With the rotameter these difficulties disappear. The wild and the controlled secondary receivers are mounted back to back in a single instrument case directly connected through an adjustable linkage by means of which any ratio whatsoever may be selected. Since the calibration of both receivers is linear and the slope of both calibrations constant over practically the whole scale, a given pen movement in either instrument will represent the same percentage flow change over the whole scale, regardless of changes in flow rate and regardless of what the selected flow ratio may be. The accompanying graphs endeavor to illustrate the fundamental shortcoming, for ratio control, of the variable-head meter square-root-scale curve and the corresponding advantage of the rotameter's first-power straight-line scale.

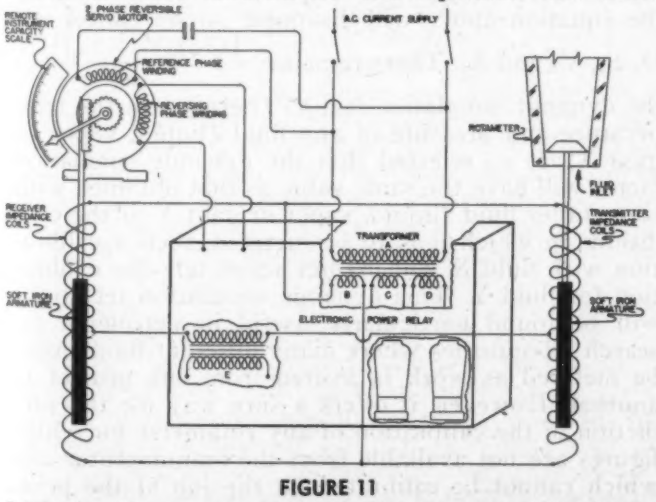


FIGURE 11
Schematic wiring diagram of Electronic Remote Transmitting System used with Rotameters. Details of amplifying and discriminating circuit are not shown.

Electronically Operated Rotameters

New electronic developments open up new possibilities in the measurement of the minute flows of additives which have had to be controlled manually or by metering-pump systems. Though the variable-area meter has long been used to indicate the "tear-drop" flows, operation of any industrially satisfactory automatic controller imposed too much force on the tiny floats which were required for low flow measurement. Now the old impedance-bridge type of transmitting system has been redesigned to produce a force only one thousandth as great as previously and has been equipped with an electronic system which detects and amplifies the current flow across the center tap of the bridge when the primary and secondary impedance coils are not in balance. The electronic system further has the ability to discriminate between the two possible directions of the unbalance and energize the reversing phase of a two-phase servo-motor so that its phase is either ahead or behind that of a reference phase winding and the motor turns in the proper direction to rebalance the bridge circuit. Standard pneumatic control systems are then operable from the secondary instrument to position small diaphragm-motor valves. Thus it is

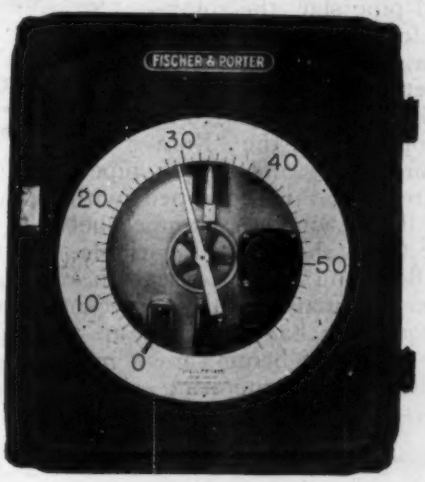


FIGURE 12
Electronic Servo-Motor Operated Remote Instrument

FIGURE 13
Tiny float and armature used for actuating the remote instrument.

now possible to use floats which weigh less than a gram, inclusive of their magnetic armature wire, and to have no effective load imposed upon them by the impedance-bridge circuit. This permits automatic control of flow rates as small as 10 cc per minute. Note that the electronic system uses no slide wire.

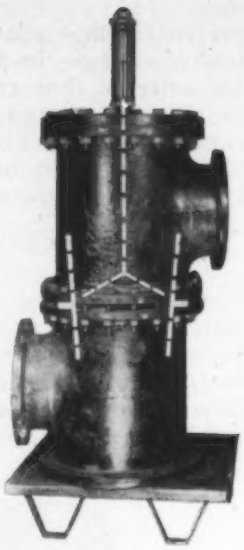


FIGURE 14
Rotasleeve with 20-inch pipe connections.

Large Capacity Rotasleeves

In the past there have been cases where it was desired to take advantage of some one of the rotameter's exclusive characteristics but, due to large capacity and pipe size, the rotameter was found to have a cost disadvantage relative to the orifice meter. However, this situation has been eliminated by the development of the "Rotasleeve" for 5-inch and larger pipe sizes. In it a variable-area tube is equipped with a horizontal mounting flange which is clamped between standard pipe tees. Only the flanged variable-area tube, the metering float and a sight extension are supplied by the instrument manufacturers. Recently the use of these Rotasleeves has extended up to 20-inch pipe size, a 20-inch unit being shown in Figure 14 with the location of the metering sleeve indicated by superimposed lines.

"Bubbler" Service Rotameters

At the other extremity of size the dimensions of flow-rate meters are being reduced to new standards of compactness. The meter shown in Figure 15 is used for such services as measuring purge line flows and air supply to pneumatic instruments in place of

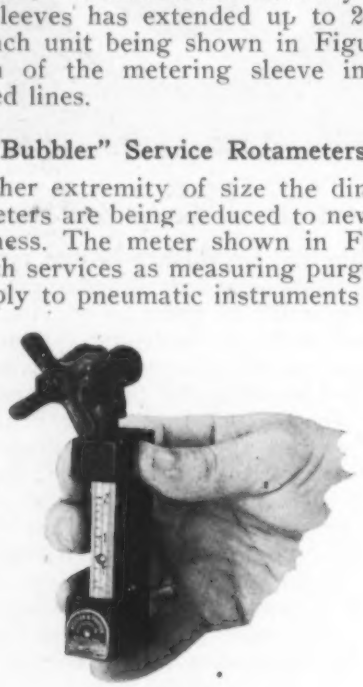


FIGURE 15
A small Rotameter for "Bubbler" service.

"sight-feed bubblers." It occupies a frontal area of only 2.6 square inches (without the valve) on a panel board and any desired number can be nested compactly body to body along a panel on a mounting rod without the loss of any panel space between the individual units. It contains no liquid (as is required in sight feed bubblers) and measures flow rate accurately rather than counting bubbles approximately.

No Pulsating Flow Problem

The use of rotameters offers a complete solution to the problem of measuring pulsating flows from slow or high-speed reciprocating pumps. Because of the linear calibration the reading averaged by eye from a pulsating float is very nearly the true reading whereas the arithmetic mean of a pulsating variable-head meter reading is not the true mean of the flow-rate variations. However, the pulsations can be eliminated by attaching to the float a rod carrying a piston and hanging it into a dashpot tube. An open valve in a bypass around the dashpot is then slowly throttled down till the dashpot action becomes sufficiently strong to reduce float pulsation to a slight vertical "quivering" which is permitted to remain as a sure indication of freedom of movement of the float. The mechanically averaged arithmetic mean readings obtained by means of the dashpot action correspond to the arithmetic progression of the scale readings.

Dynamic Simulation Technique For Transposition of Calibration Between Fluids

In closing there should be discussed a rotameter characteristic which was pointed out by A. P. Colburn and E. M. Schoenborn as far back as 1939* but which has been generally neglected and disregarded although it makes possible the calibration of rotameters with any other fluid than that with which they are to be used even though the metering floats are not of the viscosity-immune constant-flow-coefficient type and even though only a gas be available to calibrate a liquid rotameter (or vice versa). To show how this transposition of fluids can be carried out we first arrange to plot the rotameter characteristic curve not as flow coefficient C (ordinate) versus Reynolds number (abscissa) but as C versus Reynolds number divided by C. From the rotameter equation given earlier we can write

$$C = \frac{Q}{A_w \left[\frac{2g\,V_f\,(\gamma_f - \gamma_w)}{A_f\,\gamma_w}\right]^{1/2}} = \frac{V}{\left[\frac{2g\,V_f\,(\gamma_f - \gamma_w)}{A_f\,\gamma_w}\right]^{1/2}}$$

then:

$$\frac{Re}{C} = \frac{V\,\gamma_w\,D}{\mu} \times \frac{\left[\frac{2g\,V_f\,(\gamma_f - \gamma_w)}{A_f\,\gamma_w}\right]^{1/2}}{V}$$

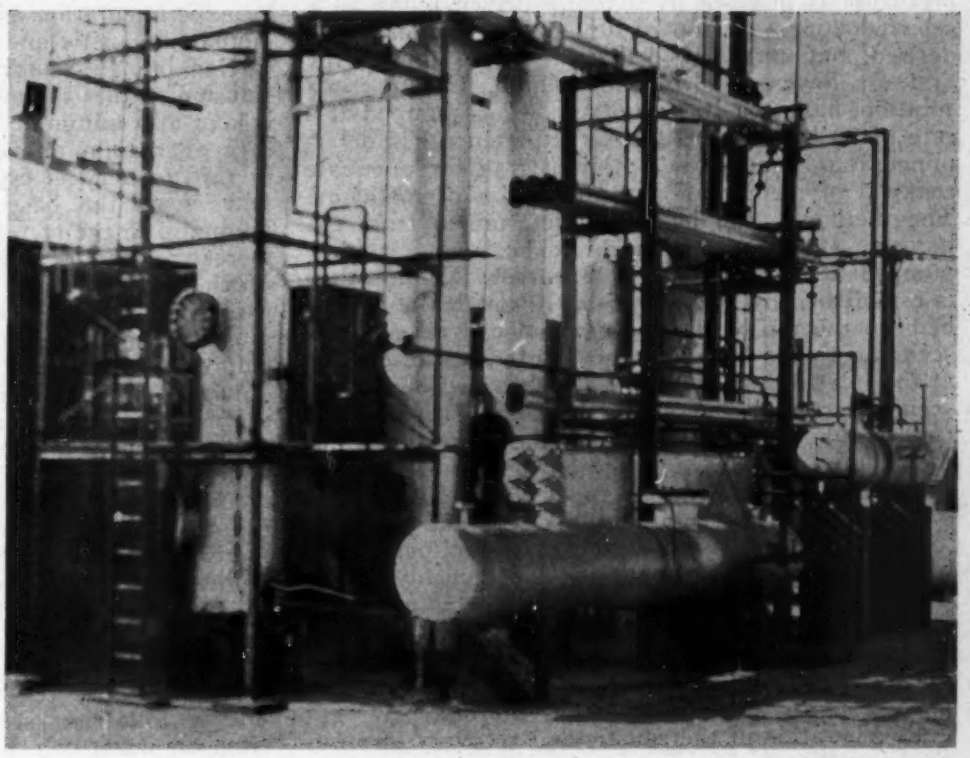
It will be seen that V (velocity), equivalent to flow rate, cancels out and we are now in a position to plot values of coefficient C, provided the physical characteristics and measurements of the fluid and rotameter are known, without determining flow rate as would be necessary if Re were used instead of Re/C. If in the case of a given rotameter we wish to predict the calibration for one fluid from that of another fluid by comparing readings height for height then all the factors determined from physical measurements in the equation above will disappear so we cancel out D, 2g, V_f and A_f. There remains $\frac{[(\gamma_w)(\gamma_f - \gamma_w)]^{1/2}}{\mu}$, the dynamic simulation factor. Therefore, if the temperature and pressure of any fluid (liquid, vapor or gas) X are so selected that the dynamic simulation factor will have the same value as that obtained with some other fluid (liquid, vapor or gas) Y at the conditions in which it is to be metered, then a calibration with fluid X will predict accurately the calibration for fluid Y. This dynamic simulation technique will be found particularly useful in petroleum research laboratories where many different fluids must be metered as work is shifted from one project to another. However, it offers a sure way for the prediction of the calibration of any rotameter for which figures are not available from the manufacturer and which cannot be calibrated on the job at the point where it is to be installed.

* "The Flow Mechanism and Performance of the Rotameter," Transactions A.I.Ch.E., Vol. 35, No. 3.

Michigan Installation Effective in

Removal of Mercaptans From Gasoline

THE Old Dutch Refining Company, refining Michigan crudes, encounters difficulties caused by degradation of gasoline due to sulfur compounds, particularly mercaptan. The current procedure is to process available Michigan crudes by topping for the production of a low-end-point straight-run gasoline, naphtha and distillates. In addition to the production of a light naphtha, or heavy gasoline, the company has access to a crude oil (distillate) produced at Evarts, Michigan, which is essentially a high-end-point, water-white gasoline. By combining refinery-produced naphtha with this "white" oil as a charge to the reformer unit, gasoline possessing all of the specifications of commercial motor fuel can be obtained, but containing mercaptan and a small percentage of hydrogen sulfide.



Base of mercaptan extraction plant in Old Dutch Refining Company refinery at Muskegon, Michigan

The processing equipment other than the topping unit, originally was installed for vapor-phase cracking, but later redesigned so it can be operated as a two-coil cracking plant, or as a naphtha-reforming unit. But in order to meet demand for a high-octane gasoline, the cracking plant has been operated as a reforming unit, charging a variable blend of naphtha and the white oil produced at Evarts.

The product of the reforming unit may be controlled as to octane and other specifications by varying the blend of the charge and adjusting the reforming plant heat, pressure and time. Normal operating proceedure is calculated to produce a maximum yield of high-octane fuel. Distillate from the reformer is condensed and stabilized to seasonal vapor pressure. Uncondensed vapors from the stabilizer are treated with absorbing oil to recover desirable fractions, while vapors from the distillate receiver are processed to convert butylenes to polymer gasoline. Reformed gasoline, stabilized to the required vapor pressure, and poly gasoline are then treated for the removal of hydrogen sulfide and mercaptan.

The mercaptan-extraction plant is a Unisol unit employing an aqueous solution of caustic soda and methanol. The unit contains five separate steps in series, all operated and controlled by flow meters and recorder-controlling instruments without requiring additional personnel.

Since the raw reformed product is processed to reduce the vapor pressure, the stabilizer column is operated at 175 psig, which is sufficient to pass the product to the desulfurizing unit without pumping. The first step in the process is to direct the flow of the product from the stabilizer to a caustic prewash column to convert the hydrogen sulfide to sodium sulphate, which is retained in the caustic wash. The wash vessel is a vertical column equipped with inlet nozzles and partially filled with carbon rings. The solution is used as a batch operation, not recirculated, and when low available caustic is observed upon analysis, is dumped and a fresh batch pumped into the column. Since the hydrogen sulfide contained in the distillate is low, the charge of caustic soda solution is carried at about 15 Be' gravity, and does not require frequent inspection.

The distillate, or reformer gasoline, is maintained at controlled temperature, and flows directly at system pressure from the prewash caustic column to the mercaptan extractor. The temperature of the gasoline is maintained between 95 and 100° F. for optimum extraction of mercaptan and a minimum loss of methanol. The mercaptan extractor is a column 2½ feet outside diameter and 73 feet high, divided into four equal compartments filled with 1-inch carbon rings resting upon steel grating. Between each bed of rings and at the top of the column are 3-foot

spaces and a 7-foot open space at the base. Spray nozzles are installed in each of the three bed-separating spaces for the introduction of methanol, and one at the top for the introduction of the aqueous solution of caustic soda.

Gasoline at a current rate of 1400 barrels per day is released from the stabilizer base through the caustic pre-wash to the extractor column, entering through an inlet nozzle at the base of the tower. The caustic solution is pumped by a motor-driven proportioning pump located in the control room, to the inlet nozzle at the top of the extractor column. Methanol is pumped by a motor-driven pump to a selected nozzle on top of any of the bed divisions desired, but currently delivered to the top of the first, or lowest bed in the column. Caustic soda solution, having a Be′ gravity of 48° is pumped at a rate of 90 barrels per day, as experience has determined that this is the optimum rate for adequate removal of mercaptan and absorption of methanol. The methanol rate has been determined from experience and experimentation to be adequate when pumped at a daily rate of 19 barrels. Counter-flow is established by gasoline rising through the packed beds to the top of the column and caustic soda descending to the base. Methanol entering the column at the inlet nozzle is selectively absorbed by the descending caustic soda, and the combination of the two liquids effectively extracts the mercaptan. The mercaptan content of untreated gasoline entering the mercaptan column averages .01, while treated gasoline leaves the column with a content of .002 to .000. Gasoline which may indicate a small percentage of mercaptan at the sample cock is quickly sweetened by oxidation in the pipe line or immediately after entering storage. Many samples of gasoline indicate that, if the mercaptan is around .002 to .001 at the treating-plant sampling cock, this same gasoline is consistently sweet immediately thereafter.

Regeneration of the caustic and recovery of methanol together with separation of the mercaptan from the methanol require following steps in the process, all of which are continuous and automatically controlled. The combined solution of caustic and methanol, plus removed mercaptan together with treating solution separated from the gasoline stream is a horizontal vessel, termed the caustic settler, flows by pumping to the caustic-stripping column of the unit. Preheating of the solution is obtained in interchanger units to obtain an inlet temperature to partially liberate methanol and mercaptan from the caustic. The caustic stripper is a vertical column 30 inches by 38 feet high, having a 20-foot section filled with 1½-inch carbon rings. A steel grating holds this bed in place above and below, and a 4½-foot bed of like filling is installed at the top of the stripper, leaving a 2-foot space clear above for vapors to collect and leave the column. The preheated treating solution enters the column above the 20-foot section of rings and flows downward for separation of methanol-mercaptans and the caustic solution.

Laboratory Inspection of Reformed Gasoline

	Charge to Treater	After Treater
Gravity	60.2	59.8
IBP	100	108
5 percent over	132	131
10	149	149
20	178	178
50	234	274
90	315	314
95	344	338
E.P.	370	368
Vapor pressure	8.4	8.0
RSH	.01	.002—.000
ASTM Octane Clear	68.3	68.8
ASTM Octane 3 cc TEL	78.2	80.3

Caustic Regenerator

The caustic-regenerator column is operated with superheated steam introduced in the base in quantities sufficient to maintain a constant gravity in the caustic solution of 48° Be′. Steam obtained from the refinery boiler is superheated in a small direct-fired unit to deliver a temperature between 650 and 700° F. A balance of heat with superheated steam is obtained by experimentation and frequent laboratory tests of methanol, caustic and treated gasoline. Operation is carried out to remove methanol from the caustic solution so that the mercaptan content of the caustic is kept at a minimum, and that the gasoline will not carry mercaptan above the required minimum, which in this refinery is essentially zero content. Caustic flows from the base of the regenerator back, by pumping, to the mercaptan extractor, and methanol-mercaptan vapors pass from the column through an overhead vapor line. With the methanol-mercaptan vapors is always a quantity of water vapor that must be removed from the composite vapor stream before the methanol is ready for re-use. This is obtained in a methanol separator, and precooling of the methanol-mercaptan vapors. Mercaptans and the water condense in the coolers and subcooler sections and accumulate in a horizontal vessel, carrying the methanol in dilute solution with the water. A wier in one end of the liquid receiver allows the liquefied mercaptan to flow to disposal. Water containing the methanol is released from the liquid receiver through a liquid-level controller so that sufficient water-methanol remains in the receiver to cover the bulk of the vessel, in order that mercaptan will not follow downward with the water-methanol. In the immediately following step, the methanol is separated from the water in a concentration desired for efficient operation by fractionating the methanol overhead in a column 2 by 33 feet, containing a 26-foot bed of carbon rings, 25 feet of which are ½-inch and a top layer 1-foot thick of 1-inch rings. Water-methanol is fed through a 1-inch line to the column 10 feet below the top of the rings. Water is drained automatically from the base of the column with liquid-control instruments, while the methanol vapors pass overhead to water-cooled condensers and to a receiver from which it is pumped back to the system as liquid. Liquefied methanol is pumped back to the rectifying column in quantities to separate water vapor by condensation in order that the methanol distillate is maintained with as high a concentration as practicable, which should be between 95 and 100.

One specific correction has recently been made in the design of the mercaptan extractor that has materially increased its efficiency. The original layout provided for a 3-inch outside downcomer connected to a flanged opening 20 feet from the base and set into the column at the juncture of the bottom head and the shell. This downcomer was suspected of draining caustic-methanol solution from the column so that contact was impared in the lower 13½-foot bed of rings. Removal of this pipe insured the extracting solution to flow downward through the bottom bed of rings so that the gasoline would be washed with solution throughout its travel from base inlet to top outlet. A more effective removal of mercaptan has been observed since the correction.

Basic Petroleum Chemistry For Refinery Employes

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Chapter VIII

PETROCHEMICALS

THERE has been a gradual evolution of petroleum refining, since World War I, into a chemical industry. The change has been rapid during the past six years as a result of World War II. We have given the name "petrochemical" to that part of the industry which is concerned with the problem of producing specific chemical compounds from petroleum.

There was practically nothing known about the chemistry of petroleum when the Drake well was drilled 87 years ago. Six years later, in 1865, Kekulé published his concept of the structure of certain hydrocarbon molecules—particularly the arrangement of the carbon atoms in the benzene molecule. This was the key which unlocked the door to the chemical developments which today enrich the lives of all.[1,2,3,4]

Until 1903 the outstanding chemical developments were in the field of synthetic dyes. Synthetic silk-like fibers, resins and thermo-plastic materials came next. All of these found the necessary starting and intermediate materials in certain fractions of coal tar. We were too busy making gasoline, fuels and lubricants for the rapidly expanding automobile industry to realize that petroleum was a far better source of organic chemicals.

Fortunately a few farsighted chemists had envisioned an organic chemical industry established upon the use of petroleum as raw material. They had for decades silently accumulated the technical knowledge necessary for the recent rapid growth of the petrochemical industry. This knowledge was available at the beginning of the recent war, when we found it necessary to plunge into a program to synthesize millions of tons of organic chemicals annually. By far the greatest tonnage during the war was, and is at this time, being used in the manufacture of synthetic rubber, aviation gasoline, solvents and plastics. A relatively small quantity of petroleum chemicals are being used in miscellaneous products such as explosives, textile fibers, anesthetics, detergents, drugs and intermediates.

Synthetic Rubber

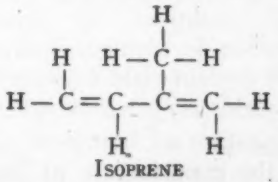
Natural rubber was in use on this continent about three thousand years ago. There is evidence of its use by the Mayas of Central America and later by the Aztecs of Mexico, and still later by the 16th century Spanish invaders led by Hernando Cortez. Despite its reputation as an essential and useful material, until a few years ago we were entirely dependent upon distant sources of supply. Without rubber, or a suitable substitute, we might have lost the war to the Axis powers.

In 1941 we actually produced 17,000 long tons of all types of synthetic rubber. When the supply of the natural product was cut off by our war with Japan, our annual requirements were well over a half million long tons. Our Allies were requiring another half million long tons.

In an incredibly short time some 20,000 American chemists, engineers, and workers were producing as much rubber as 400,000 natives could tap from 200 million trees, on plantations which had taken over a half century of cultivation and development. By the close of the second year of the war we were making synthetic rubber at the rate of about one half million long tons per year, and at the close of the war we had reached a production rate of almost a million long tons per year.

The accomplishment of this task will perhaps stand for many generations as the outstanding achievement of the age in the fields of chemistry and engineering. It is all the more remarkable when we consider that the search for suitable rubber substitutes had been going on for almost a century.

Michael Faraday demonstrated in 1826 that rubber was composed of five atoms of carbon and eight atoms of hydrogen. Later the structure of the primary ingredient of natural rubber was learned and named *isoprene*. The structure was established as:



ISOPRENE

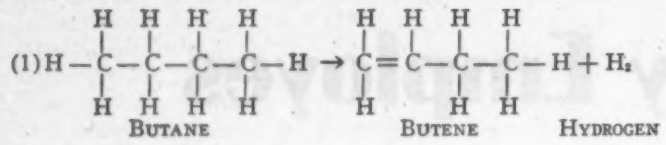
This basic knowledge was followed with contributions from chemists in several countries, notably Germany, Russia, England and America. It was from these contributions that the wartime synthetic rubber program was evolved.[5]

Chemically, the synthetic rubbers or *elastomers* may be classified into five types, all of which may require one or several petrochemicals.[5,6]

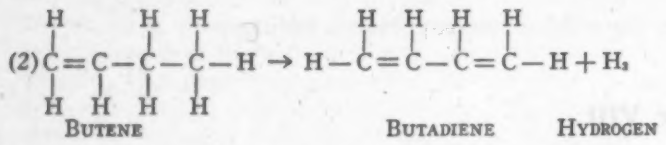
(1) *Butadiene polymers and copolymers* are made by the polymerization of butadiene or the copolymerization

of butadiene with some other unsaturated hydrocarbon such as isoprene or isobutylene.

In the petrochemical industry butadiene is made by the dehydrogenation of butane and butene. Both thermal and catalytic processes are used. The reactions are:



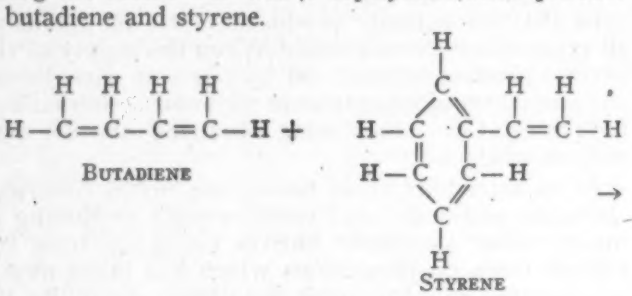
(1) Butane → Butene + Hydrogen



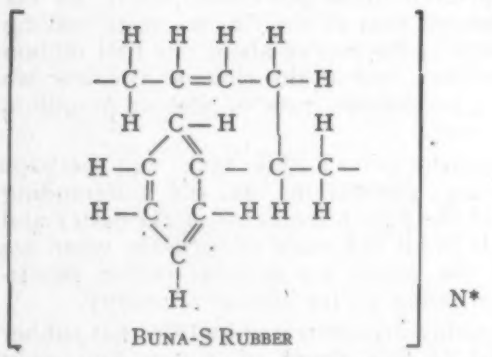
(2) Butene → Butadiene + Hydrogen

The polymerization of butadiene using sodium as a catalyst, yields a rubber-like material which can be vulcanized. It is known as buna rubber, taking its name from the first two letters of butadiene and natrium (sodium).

Most of the synthetic rubber made in this country during the recent war was a copolymerization product of butadiene and styrene.



Butadiene + Styrene →

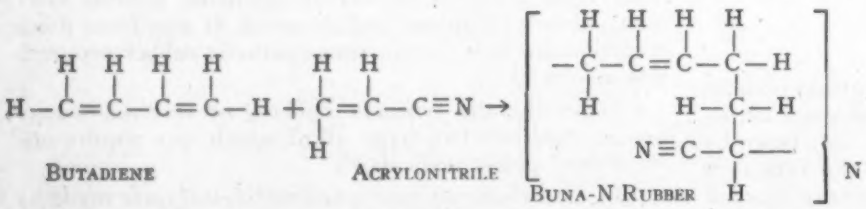


Buna-S Rubber

* The "N" represents a whole number indicating repeating structures.

Ethylene is a byproduct of refinery cracking operations. When this unsaturated compound is acted upon, under controlled conditions, by hypochlorous acid, *ethylene chlorohydrin* is formed. Sodium cyanide and the ethylene chlorohydrin yield *hydracrylic nitrile*. This latter compound yields *acrylonitrile* when dehydrated.

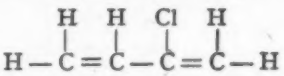
The copolymerization of butadiene and acrylonitrile is the basis for the manufacture of Buna-N synthetic rubbers. The final reaction is represented as follows:



Butadiene + Acrylonitrile → Buna-N Rubber

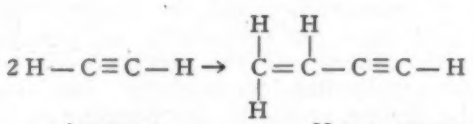
The polymerization of dimethylbutadiene was the source of *methyl* rubber used to some extent by the Germans in the first World War. Copolymerization with normal butadiene, isoprene, styrene, etc., are also possible.

(2) *Substituted butadiene polymers.* A number of rubber-like polymers are possible by the combination of the butadiene molecule with chlorine as well as styrene and acrylonitrile. Chloroprene is a butadiene molecule having a chlorine atom in place of one of the hydrogen atoms. Its structure is as follows:



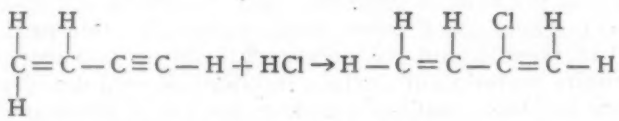
Chloroprene

This compound is the chief ingredient of *neoprene*, and while it may be produced from petroleum acetylene, its commercial production has been chiefly from lime, coke and hydrogen chloride. The lime and carbon are combined in a high-temperature electric furnace to form calcium carbide ($Ca C_2$), which reacts with water, liberating the acetylene gas. The polymerization of two molecules of acetylene yields one molecule of *vinylacetylene.*



Acetylene → Vinylacetylene

The reaction of hydrogen chloride with vinylacetylene yields the compound known as *chloroprene.*



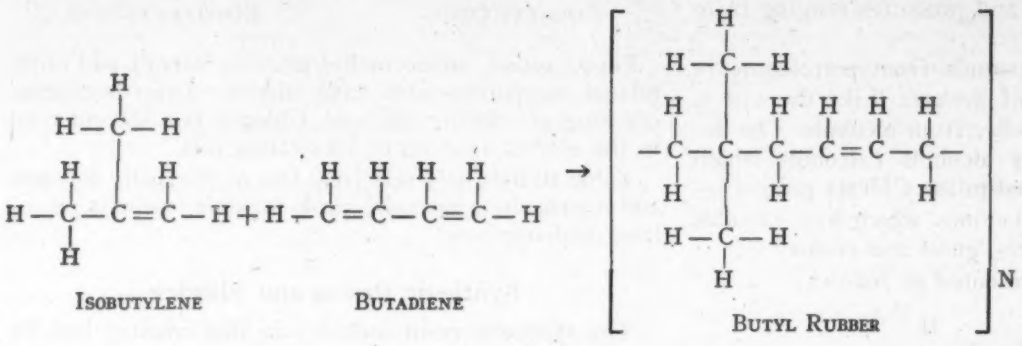
Vinylacetylene + Hydrogen Chloride → Chloroprene

These reactions were thoroughly investigated prior to 1932 by Father Julius Nieuwland of the University of Notre Dame, Department of Chemistry, and were developed commercially by E. I. du Pont de Nemours & Company to yield the synthetic rubber *neoprene.*[5]

(3) *Butene polymers and copolymers.* Isobutylene is also a byproduct of refinery cracking operations. It is prepared commercially by the isomerization of normal butane followed by dehydrogenation. Butyl synthetic rubber is made by the copolymerization of about 98 percent isobutylene with other unsaturated hydrocarbons such as isoprene and butadiene. This rubber is sometimes referred to as "bathtub rubber." The copolymerization takes place at low temperature in the presence

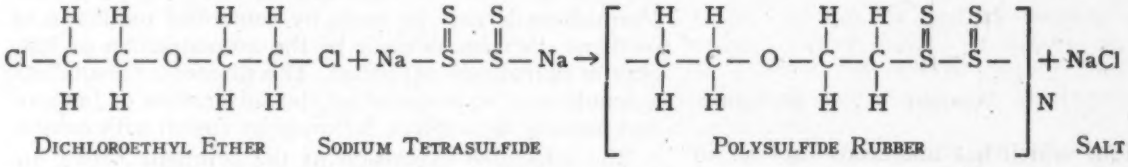
of catalysts such as titanium tetrachloride, boron fluoride and aluminum chloride.

The reaction of the isobutylene with butadiene is represented below:



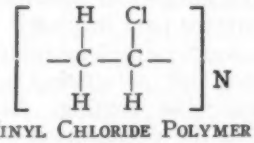
ISOBUTYLENE BUTADIENE BUTYL RUBBER

(4) *Organic Polysulfides.* Organic dihalides such as ethylene dichloride react with sodium polysulfide to form rubber-like elastomers. One commercial synthetic which belongs to this group is represented below:



DICHLOROETHYL ETHER SODIUM TETRASULFIDE POLYSULFIDE RUBBER SALT

(5) *Vinyl Chloride Polymers.* Vinyl chloride is made by the reaction of acetylene and hydrogen chloride, the direct chlorination of ethylene to the dichloride followed by dehydrochlorinization with alcoholic caustic, or by vapor-phase cracking of ethylene dichloride. Upon polymerization the polymer has the structure represented below:



VINYL CHLORIDE POLYMER

Since the polymer does not have double or triple bonds in its structure, it cannot be reacted with sulfur (vulcanization).[7,8]

Aviation Gasoline

A number of petrochemical processes were developed during the past ten years for the purpose of making specific organic compounds from petroleum for use in aviation fuels. Some of the more important of these processes were discussed in Chapter VI of this series, "Manufacture of Aviation Gasoline Components." Table 8 in Chapter VI lists some of these petrochemicals and their important properties.

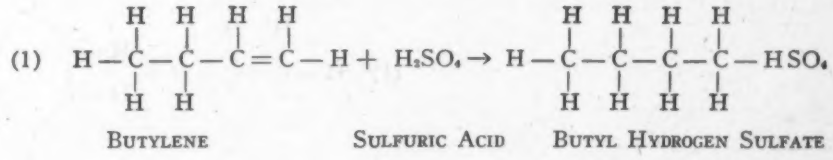
Solvents

The petroleum refiner has for many years supplied industry with a variety of highly-refined hydrocarbon solvents for use in dry cleaning, the manufacture of synthetic fibers, printers ink, rubber goods, paints, lacquers and plastics. Most of these solvents have been closely-fractionated cuts taken directly from selected crude oils.

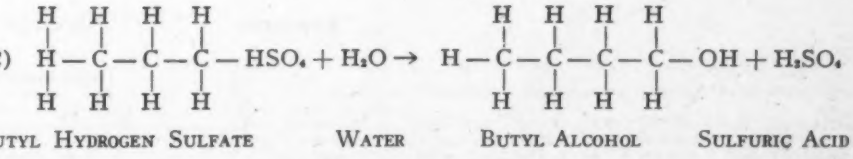
Mass production methods of many manufactured items, particularly automobiles, were greatly facilitated by the introduction of fast-drying solvents and thinners to paints and lacquers. Petroleum was found to be the best and most economical source for some of these fast-drying solvents and thinners.[7,9]

Butyl acetate from petroleum involves the conversion of butylene to butyl alcohol followed by esterification of the alcohol with acetic acid. The reaction of an alcohol with an acid yields a compound known as an ester, and the reaction is called esterification.

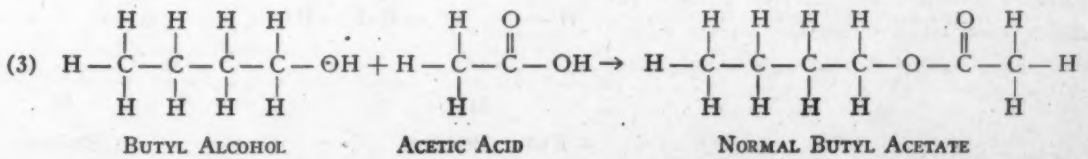
The reactions are:



BUTYLENE SULFURIC ACID BUTYL HYDROGEN SULFATE



BUTYL HYDROGEN SULFATE WATER BUTYL ALCOHOL SULFURIC ACID

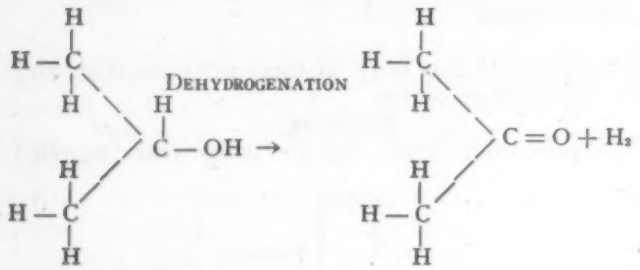


BUTYL ALCOHOL ACETIC ACID NORMAL BUTYL ACETATE

It is possible to prepare certain esters by direct esterification of unsaturated hydrocarbons with organic acids. For example, butyl acetate may be prepared by counter-current contacting of butylene with acetic acid, containing a small quantity of sulfuric acid, at temperatures of 120° F. to 150° F. and pressures ranging from 100 to 180 psi.

A similar group of compounds from petroleum are represented by the family of *ketones*. Like the esters, they are also prepared, usually, from alcohols. The dehydrogenation of secondary alcohols (alcohols which have two carbon atoms linked to the CHOH group) results in the formation of ketones which are valuable solvents for cellulose lacquers, gums and resins.

A typical reaction is represented as follows:

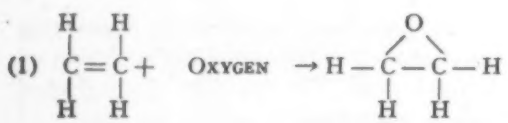


ISOPROPYL ALCOHOL ACETONE HYDROGEN

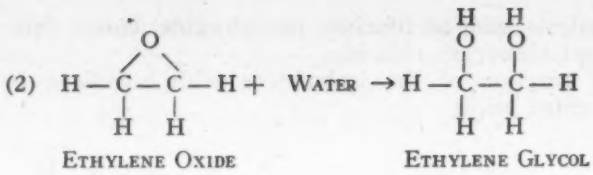
Acetone is a ketone which has numerous uses as an industrial solvent.

We have seen in Chapter V of this series, how low-octane petroleum naphthas are converted by the Hydroforming process into aromatic compounds such as benzene, toluene, and the xylenes. These petrochemicals have become very important as ingredients for aviation gasoline, intermediates in chemical synthesis, and as solvents.

Ethylene is also used for the preparation of the popular antifreeze solution, *ethylene glycol*. The reactions are:



ETHYLENE ETHYLENE OXIDE



ETHYLENE OXIDE ETHYLENE GLYCOL

Glycol esters, dichloroethyl ether (*chlorex*) and other related compounds also have ethylene from petroleum cracking as starting material. Chlorex is a solvent used in the solvent refining of lubricating oils.

Table 10 lists only relatively few of the many solvents and intermediate petrochemicals possible from six petroleum hydrocarbons.

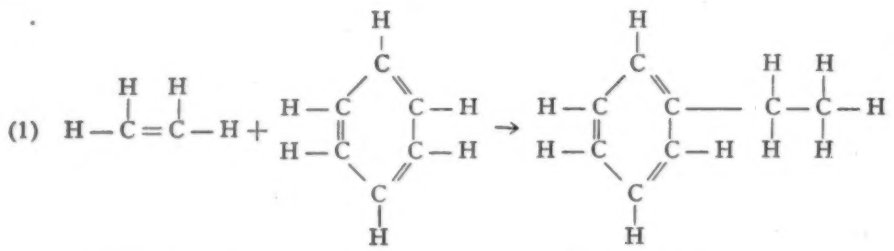
Synthetic Resins and Plastics

The synthetic resin industry in this country had its origin in 1909 with the successful experiments of Dr. Leo Backeland. His experiments gave birth to the Bakelite industry. Formaldehyde, benzene, phenol, cresols, xylenols, urea and melamine, the intermediates used in the Bakelite industry, originally were derived from coal. Now, at least some of them are derived from petroleum. Formaldehyde may be made by controlled oxidation of methane. Benzene is made by the aromatization of low-octane petroleum naphthas. The phenols, cresols and xylenols may be prepared by the sulfonation of benzene and benzene derivatives, followed by fusion with caustic.

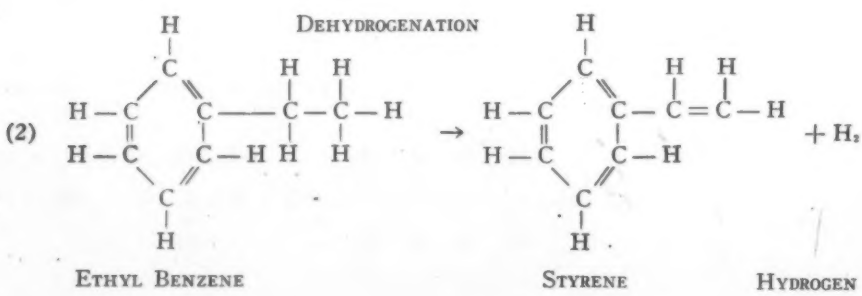
The scheduled expansion of the synthetic plastic industry calls for the expenditure of $150,000,000 as soon as necessary materials are available. The demand at the present time for plastics of all kinds is at least three times greater than the supply.[10] The petroleum industry will supply a large volume of the intermediates necessary to meet this great demand.[11] We have mentioned the use of ethylene in the preparation of synthetic rubbers, solvents, and a number of intermediate products. It also plays an important part in plastic materials.

The polymerization of ethylene molecules is the basis for the preparation of *polythene*, a plastic material used for electrical-cable coating. As previously discussed, it is used in the preparation of rubber-like materials from polyvinyl chloride and polyvinylidene chloride.

The reaction of ethylene with benzene followed by dehydrogenation, forms styrene, one of the ingredients of Buna-S rubber. The reactions are:



ETHYLENE BENZENE ETHYL BENZENE



ETHYL BENZENE STYRENE HYDROGEN

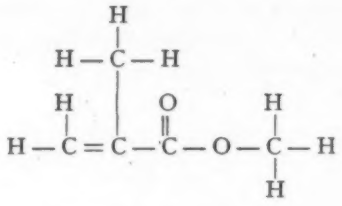
TABLE 10

Petrochemicals Possible from Six Hydrocarbons

From Methane	From Ethane	From Ethylene	From Propane	From Butane	From Isobutane
Methyl alcohol	Ethyl alcohol	Ethylene chloride	Paraldehyde	Secondary butyl alcohols	Tertiary butyl alcohol
Methyl halides	Ethyl halides	Ethylene glycol	Propyl alcohols	Butyl acetate	Isobutylene chloride
Methyl esters	Acetic acid	Dichloroethyl ether	Propylene chloride	Butylene chloride	Isobutylene chlorohydrin
Formaldehyde	Acetaldehyde	Ethylene diamine	Propylene chlorohydrin	Butylene chlorohydrin	Trimethyl pentanes
Carbon disulfide	Diethyl ether	Succinic acid	Isopropyl halides	Polymer alkylates	Sulfur dioxide resins
Hydrogen sulfide	Ethyl esters	Sodium polysulfide	Isopropyl ether	Sulfur dioxide resins	Tertiary butyl phenol resins
Nitromethane	Metallic ethylates	Vinyl acetate	Isopropyl acetate	Butadiene	Polymer alkylates
Nitroform					

When styrene is polymerized, a transparent plastic, polystyrene, is formed. It has excellent resistance to chemicals and moisture and is being used in increasing quantities for electrical insulation in radio, radar and television equipment.

Another family of useful polymers is comprised of the *acrylic* resins. They are obtained indirectly from cracked gases. The most useful resin in this group is *methyl methacrylate* which has the following formula:



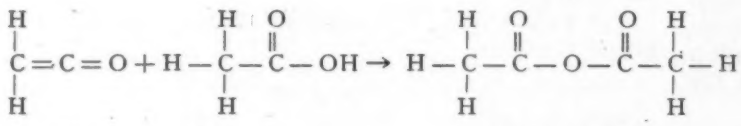
METHYL METHACRYLATE

It is produced chiefly from propylene through reactions which involve isopropyl alcohol, acetone, hydrogen cyanide, acetone cyanohydrin and methyl alcohol. Polymethyl methacrylate resins find uses in numerous applications because of their unique property of being able to bend light rays. This is a useful property in the manufacture of various shapes of surgical lights.

Acetylene and acetic acid form vinyl acetate which is copolymerized with vinyl chlorides, vinyl alcohols and other similar compounds to produce a number of waterproofing plastics useful for coating fabrics.

Miscellaneous Products

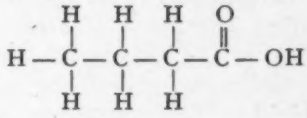
The rayon cellulose acetate industry realized its most rapid development after a method was found to make *acetic anhydride* from propylene or acetylene. The propylene is converted to isopropyl alcohol, and the alcohol is oxidized to acetone as previously explained. Acetone upon cracking yields *ketene*. The reaction of the ketene with acetic acid yields acetic anhydride, as represented below:



KETENE ACETIC ACID ACETIC ANHYDRIDE

The acetic acid may be made by the controlled oxidation of ethane.

Butyric acid, which has the formula represented below, is derived commercially from ethylene.



BUTYRIC ACID

It is used in the preparation of the synthetic fiber material known as *cellulose acetobutyrate.*

The polyamides are of particular interest to the women. Better known as *nylons,* the polyamides were the cause of numerous waiting lines not so long ago, when both men and women lined up for the purchase of nylon hose.

Ethylene again enters the petrochemical picture in the preparations known as *surface-active agents.* They possess the property of making water wetter, and for that reason are used widely as detergents, sometimes referred to as "soapless soaps."

Ethylene chlorohydrin, when reacted with ammonia, forms *mono-, di-* and *tri-ethanolamines.* When these are combined with fatty acids valuable detergents are produced.

Sulfonated petroleum hydrocarbons are finding an increasing demand as wetting agents. A number of sulfonated aromatics, naphthalenes and phenols are on the market as detergents, emulsifying agents, wetting agents and penetrants.[7]

The manufacture of explosives, lubricants, drugs, cosmetics, agricultural preparations are being developed, and many more specific uses are being found for the products of the expanding petrochemical industry. The relatively few uses, mentioned in this chapter, require a greater volume of petrochemicals than the young industry can supply at this time. There seems to be very little likelihood that the demand for them will soon diminish. There is much evidence that the demand will greatly increase.

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DISCUSSION

Pressure Vessel Design Calculations

THE attention of the ASME Special Research Committee on Vessels Under External Pressure has been called to certain errors in the paper by S. M. Jorgensen entitled "Pressure Vessel Design Calculations."

A portion of this paper is devoted to the design of vacuum vessels. The basic formula for the design of vacuum vessels using a factor of safety of 4, is given as

$$\left[1.23\times10^{6}\left(\frac{t}{D}\right)^{2}+0.45\right]\left(\frac{t}{D}\right)^{\frac{1}{2}}=\frac{L}{D}\times\frac{29\times10^{6}}{E} \qquad (12)$$

This formula is in error in that the term 0.45 should be replaced by

$$\frac{29\times10^{6}}{E}\times0.45.$$

However, since the term is usually small compared with the other term to which it is added, this error is not important.

Equation (12) is represented in Figure 3 in the form of a chart. The chart is intended to give the required thickness for a steel pressure vessel working under a vacuum at temperatures up to 1000° F. The chart may be correct for the region in which equation (12) is applicable provides it makes proper correction for increased temperatures. However, as given in Figure 3, the chart is extended to regions where equation (12) is not applicable.

The author has pointed out that equation (12) does not apply for high values of L/D, and replaces it by the Bresse-Bryan formula. However, in solving the Bresse-Bryan formula for a pressure of 15 pounds per square inch and a factor of safety of 4, the author gets.

$$t=\frac{D}{93.2} \qquad (13)$$

Equation (13) is in error, since the elastic modulus does not appear. This error is serious since the elastic modulus decreases rapidly with increasing temperature. Hence, Figure 3 is incorrect in the region of high values of L/D. It should not be used for values of L/D exceeding 12.

The author did not point out that for relatively thin vessels with small length-diameter ratios, collapse occurs under external pressure when the hoop stress reaches the yield stress. Equation (12) is not applicable in this region either, but predicts collapsing pressures that are too high. Hence, Figure 3 is incorrect in the region of low values of L/D also, and should not be used for values of L/D less than 0.2.

It is true that the values of L/D and t/D for which equation (12) is not applicable are seldom encountered in the design of vacuum vessels. Nevertheless, the limits within which the formula can be used with safety should be clearly understood if costly failures are to be prevented. If the formula is to be used in the form of a chart, similar to Figure 3, the chart should be confined to those values of L/D and t/D for which the formula gives reliable design values.

It is recommended that the charts given in the ASME Code be used for vessels under normal temperatures and that those in the API-ASME Code be used for elevated temperatures, instead of Figure 3, for the design of vacuum vessels to work at elevated temperatures.

In answer to which the author, S. M. Jorgensen comments:

The error in the transformed Wiedenburg formula (12), Figure 3, is intentional, and has also been committed by the API-ASME Code, as an examination of paragraph EW-306, Figure 27, will show. In this chart the abscissa X is directly proportional to

$$\frac{29\times10^{6}}{E}\times\frac{L}{D}$$

The omission is negligible as far as shell thicknesses are concerned, and greatly simplifies thickness calculations.

For values of L/D exceeding 12, the thickness t is proportional to

$$\sqrt[3]{\frac{29\times10^{6}}{E}}$$

The lower limit of L/D for using formula (12), Figure 3, would seem to be around 0.07 rather than 0.2.

The author agrees that formula 12, Figure 3, is applicable only within limits of

$$0.07<\frac{L}{D}<12$$

and doubts very much that there is any refinery vacuum vessel (15 psi external) in existence which falls outside these limits. Within these limits Figure 3 has proved to be a fast and time-saving tool.

Coles Levee Cycling Plant

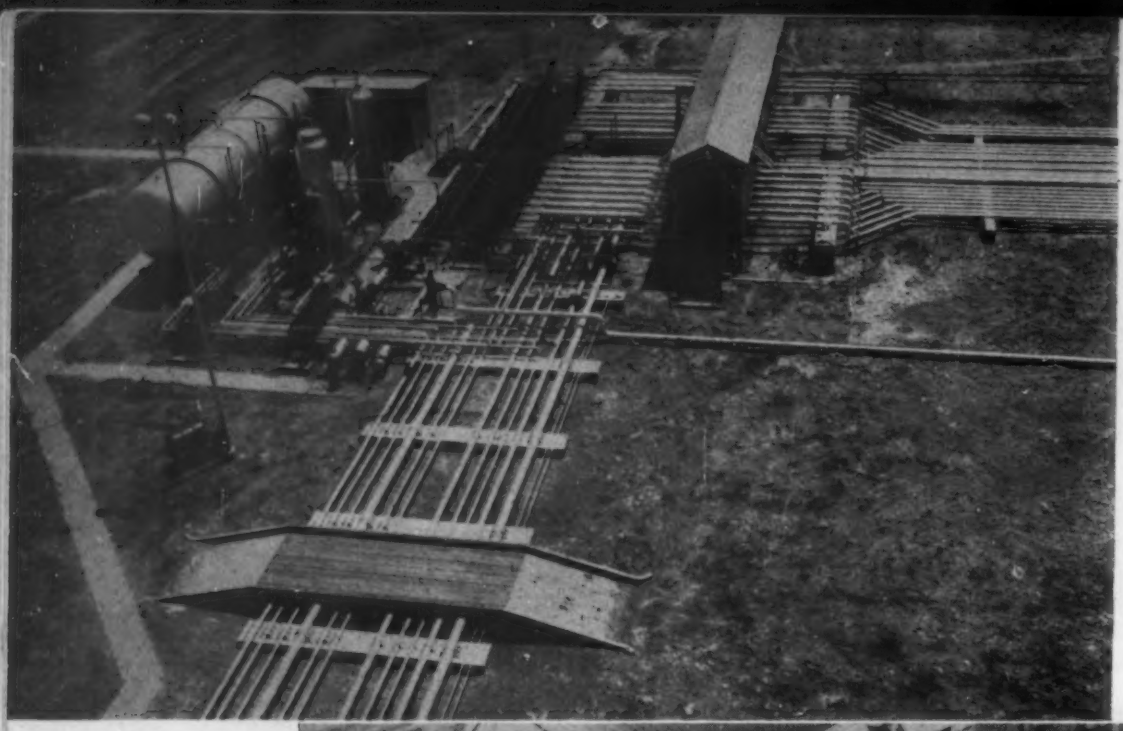
Includes Advanced Design Technique

C. C. PRYOR, Associate Editor

CORROSIVE soil conditions in the area southwest of Bakersfield, California, made it desirable to install process piping above ground in construction of the South Coles Levee Unit cycling plant, a unitization project which was put in operation last year. Design engineers incorporated a number of advancements in the plant, which includes manifolding of inlet-gas lines for centralized measurement and individual well testing at the plant; use of a new type pulsation dampener on the compressor suction and discharge lines; a plant piping design layout to eliminate vibration, expansion loops, corrosion from alkaline soil conditions, and unnecessary elevated lines; absorption of high-pressure distillate gas and low-pressure casinghead gas; instrumentation to prevent liquid carryover to the compressors, and a dual emergency shutdown system. The plant has a high operating efficiency and low maintenance cost.

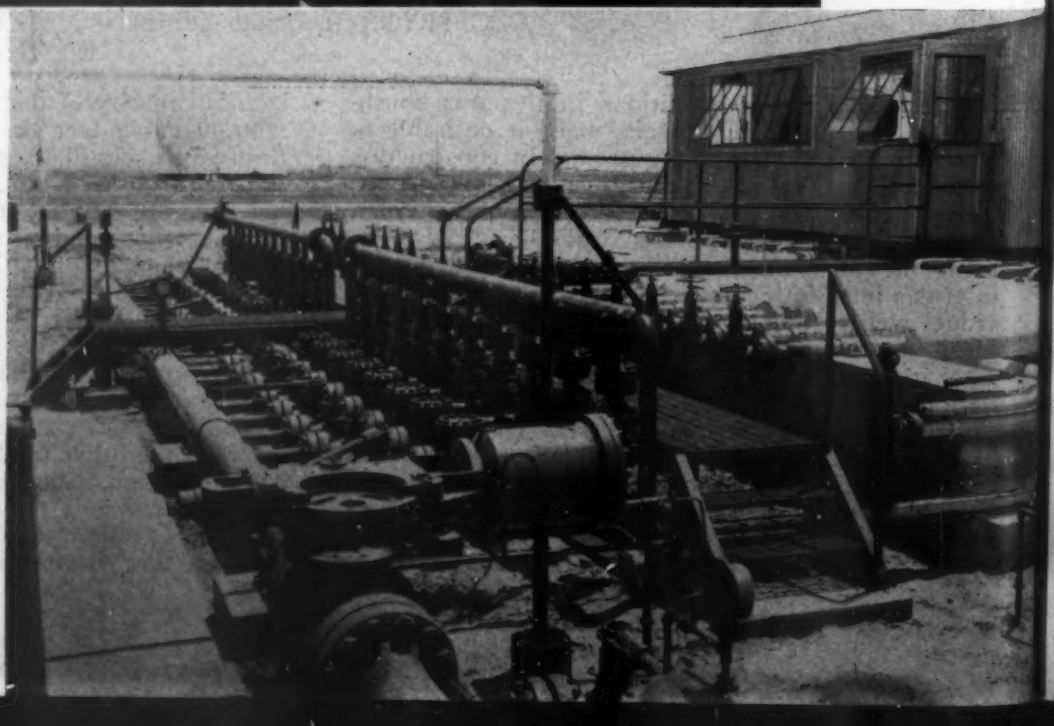
Design capacity of the absorption-oil-type plant is 60,000,000 cubic feet daily, of which 45,000,000 is high-pressure distillate gas and 15,000,000 is low-pressure casinghead gas. At present, the plant is recovering 98 percent butanes, 88 percent isobutane, approximately 30 percent propane, and all pentane and heavier fractions. Operating at capacity, the plant is expected to recover approximately 7000 barrels of all liquids daily—4650 barrels of 50° API debutanized condensate, 715 barrels of natural gasoline, 660 barrels of normal butane, 440 barrels of isobutane, and up to 600 barrels of propane. Operation is by Ohio Oil Company through a unitization agreement with the other producers in the field, Richfield Oil Corporation, Standard Oil Company of California, Tide Water Associated Oil Company, and Union Oil Company of California.

A departure from the conventional method of



Production lines from all wells in the field flow at wellhead pressure to a point near southeast corner of the plant entering at right. Test separator unit is at left of main manifold. Note large ramp in foreground which permits vehicles to cross surface lines. Center section is removable when necessary to replace or remove lines.

Interior view of meter house showing orifice recording meters measuring gas flow from each well as lines enter plant area. Steam jacketed lines below meter house prevent hydrate formation in orifice fittings.



Main manifold and test separator unit manifold for incoming production lines showing check valves, flow beans, and steam-jacketed heaters. Pressure-reducing valve maintains constant pressure on 1500-pound absorber. Master valve, piston-operated, shuts off incoming gas to plant in emergency. All lines, connections, valves, and controls are readily accessible for maintenance

metering gas at the wellhead and manifolding the inlet-gas lines in the field is represented by the metering, testing, and manifolding system employed at the South Coles Levee plant.

All inlet-gas lines from the high-pressure distillate wells enter the plant site at one point. Heavy concrete piers support the lines about two feet above the ground level, making them accessible for maintenance and inspection. Underground lines were considered unsatisfactory due to alkaline soil conditions, and overhead piping is used only in the processing area. Each inlet line is equipped with an orifice meter, steam-jacketed heater, flow bean, and check valve at the main manifold header, in which the well's effluent is commingled. Distillate-gas pressures vary from 1800 to 2600 psig and temperatures from 110° to 140° F.; however, well effluent is metered at full well-head pressure, which then is reduced to 1800 psig by the flow bean before entering the main manifold. Gas and liquid from the manifold flow through a pressure-reducing regulator, controlled by the pressure of the high-pressure absorber residue scrubber, to maintain a constant pressure on the 1500-pound absorber.

A test separator unit is manifolded to the lines from the wells and gas-oil ratios are checked by diverting the gas stream from a well into the unit. Gas to the test unit flows through cooling coils, which holds the temperature constant for the tests, to a separator operated at 1500 psig. From the separator, the gas is metered and flows to the high-pressure absorber. Raw condensate from this separator is flashed in a 450-psig separator, the gas flowing through an orifice meter to the 450-psig feed line to the 24-tray high-pressure reabsorber and the condensate flashed in a 60-psig test separator. Flashed gas is metered and flows into the casinghead gas stream to the plant at 60 psig. Liquid is accumulated and measured in the 60-psig trap, which is a horizontal vessel 7 feet by 30 feet and is capable of holding raw condensate from any well for a period of from 12 to 24 hours. From the recorded volumes and gravities, a gas-oil ratio and percent-water cut is determined and a low-temperature fractional-distillation test made on a liquid and gas sample from each well. The simplified form used by the operators is shown in Figure 1.

The composite gas sample taken of the three gas streams eliminates a separate analysis of each stream

TEST SEPARATOR ANALYSIS
S. C. L. U.

Well No..... Date.....
Test Period.....Hours Date last tested.....
Casing Pressure.....PSI. Tubing Pressure.....PSI.

	Composite Gas			60 lb Liquid				Composite Gas & Liq.		
	Mol %	Mol/D	Gal/D	Liq. %	Gal/D	Mol/D	Mol %	Mol/D	Mol %	Gal/D
C_1										
C_2										
C_3										
iC_4										
nC_4										
iC_5+										

1500 lb Gas.....MCF/D Oil Produced.....Bbl/D
450 lb Gas.....MCF/D Oil Gravity.....° API
60 lb Gas.....MCF/D Oil Cut.....% BS&W
Free H_2O Produced.....Bbl/D
Total Gas.....MCF/D Total H_2O Produced....Bbl/D = ...% Cut
Vapor Equivalent Factor for 105 Fraction of 60lb Liquid.....Cu. ft/Gal.
Equivalent Vapor from 60lb Liquid - - - - - - .....MCF/Day
Total Gas Plus Condensate Vapor, A= - - - - - .....MCF/Day
Total Gas as shown on well Meter, B= - - - - - .....MCF/Day

Correction Factor for Meter $\frac{A}{B}$ = - - - - - - - .....

Iso-Butane Ratio = $\frac{\text{Iso-Butane Produced}}{\text{Total Gas \& Condensate Vapor}}$ = D = ...Gal/Mcf

Normal Butane Ratio = $\frac{\text{Normal Butane Produced}}{\text{Total Gas \& Condensate Vapor}}$ = E.....Gal/Mcf

Iso-Pentane Plus Ratio = $\frac{\text{Iso-Pentane Plus Produced}}{\text{Total Gas \& Condensate Vapor}}$ = F = ...Gal/Mcf

Total Recoverable Product Ratio = D+E—Y = ...

Gas Oil Ratio = $\frac{\text{Total Gas}}{\text{Oil Produced}} = \frac{\text{CF}}{\text{Bbl.}}$

FIGURE 1

and is taken in absolute pressure increments that are proportional to the volume of each stream. The final absolute pressure is held below 300 pounds to minimize the effect of supercompressibility. Composition of the well effluent is determined by combining the analysis of the 60-pound liquid with the analysis of the composite gas sample. Gas-oil ratios are checked monthly on each well and fractional analyses of the effluent made only when there is a change in gas-oil ratio.

The design of the absorption system simplifies recovery of liquid hydrocarbons from the casinghead

Pipe vibrations in the 400- and 5000-pound discharge lines have been eliminated by use of special pipe clamps instead of expansion loops; frequent pier spacing; large lines resulting in gas velocities of 5 to 6 feet per second; large suction and discharge chambers, and new type pulsation dampeners.

Box-type clamps are used on first two piers on either side of the corner when a line changes direction, to permit longitudinal and lateral expansion. Spring-loaded anchor clamp permits longitudinal expansion (foreground). Note concrete piers for lines crossing lines on main piers. Spring-loaded anchor clamps effectively snub pipe vibrations, but permit necessary expansion movement and results in reducing plant repairs and shutdowns.

gas, which enters the plant at three different pressures—450 psig, 60 psig, and 6-inch vacuum. In addition to the high-pressure absorber, the plant utilizes a high-pressure reabsorber and a low-pressure reabsorber. Rich-oil streams are finally combined in a vent tank.

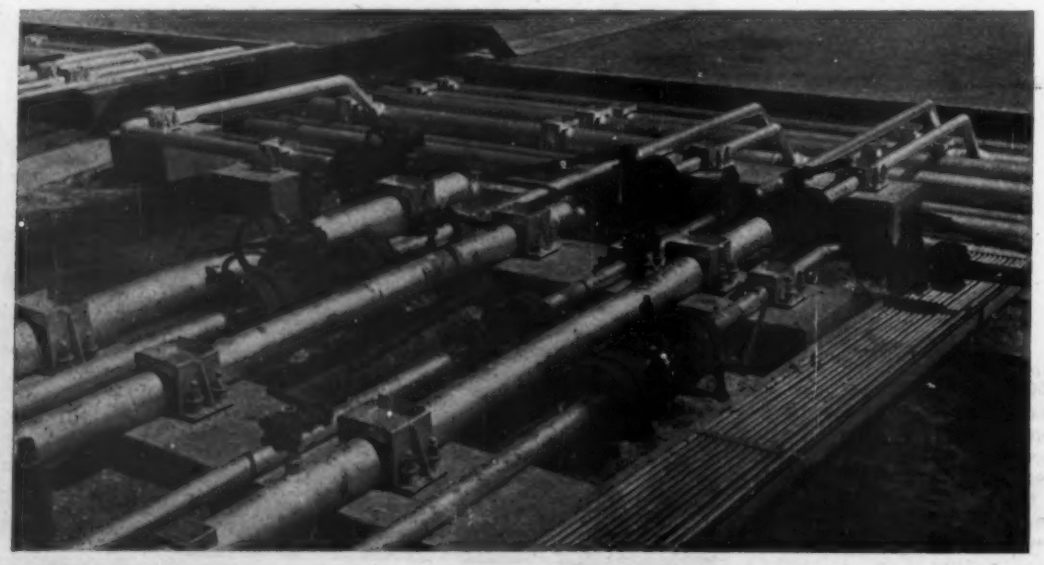
Casinghead gas at 450 psig flows through a meter into the 450-pound separator in which the raw distillate from the 1500-pound separator is flashed. Then the gas from the 450-pound separator is metered and charged to the high-pressure reabsorber. To combine the rich-oil stream from the high-pressure absorber with that from the high-pressure reabsorber, the lower section of the reabsorber was designed as a 450-pound flash chamber. The reabsorber is 5 feet in diameter by 63 feet in height and has 24 trays.

The 60-psig casinghead gas flows into the 60-pound trap tank where it mixes with the flashed vapors of the raw distillate from the 450-pound separator. This tank is used as a liquid charge tank for the crude-rectifier tower.

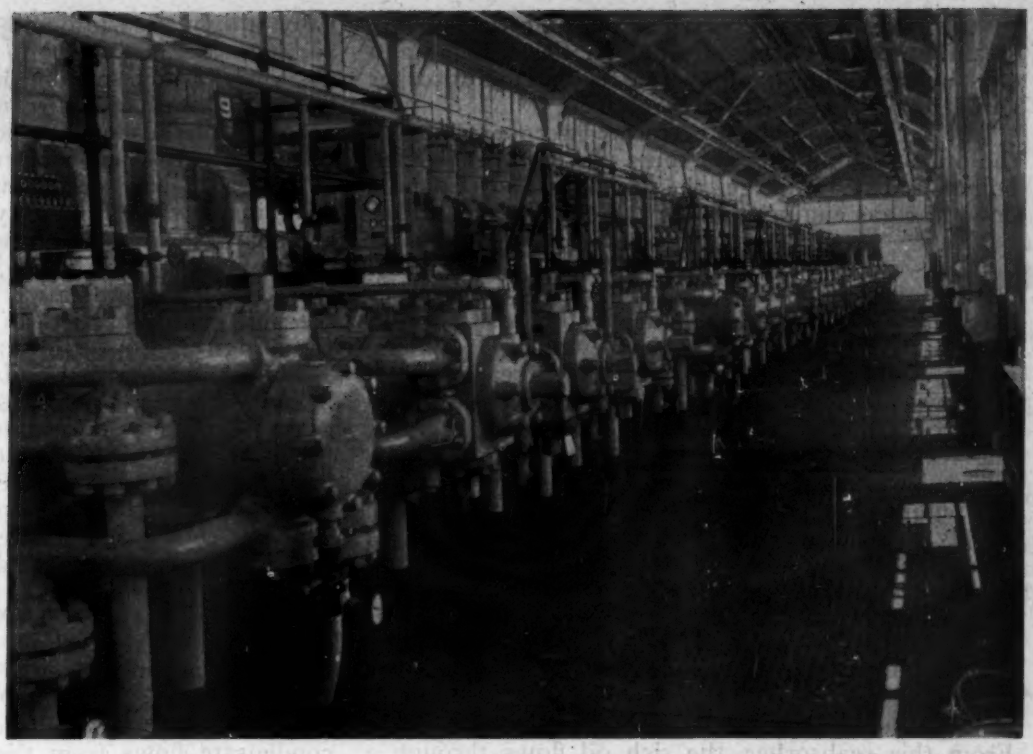
A vacuum accumulator receives the 6-inch vacuum casinghead gas from the crude-oil storage tanks in the field and from the debutanized-condensate storage tanks in the plant. After passing through two stages of compression, this gas enters the line to the low-pressure reabsorber at 175 psig. Residue gas from this reabsorber flows directly into the plant fuel system. Rich-oil from the high-pressure reabsorber is vented in a tank 5 feet by 10 feet and the vented gas charged to the reabsorber. Bottoms from the low-pressure reabsorber flow to the vent tank.

Compressor Manifolding

There are nine 600-horsepower, two-cycle, gas-engine-driven compressors in a single bank in the compressor building. Each engine has four compressor cylinders so arranged that one of the four is for 1500-pound and one or more for 4000-pound pressures, with the remaining cylinders used for low pressure. This arrangement provides a capacity balance for the high-pressure services on each compressor. The compressor system is designed to return 54,500,000 cubic feet daily of absorber residue gas to the in-



Intake and discharge lines from the compressors to the headers. Box clamps are used on pulsation dampeners in discharge lines. Manifold lines are below the level of the intake and discharge lines to provide easy accessibility. Concrete piers closely spaced support heavy flanges and valves.



Nine, 600-horsepower, two-cycle, gas-engine-driven, compressors in a single bank in the compressor building are designed to return 54,500,000 cubic feet daily of residue gas to the injection system at a pressure of 4000 pounds.

jection system at a pressure of 4000 psig. Compressor-suction pressures vary from 6 inches vacuum to 1500 psig and discharge pressures from 30 to 4000 psig.

Particular attention was given to five points in the design of the compressor piping to eliminate vibration of the lines and pulsating flow of 1500- and 4000-pound gas: (1) special pipe-line clamps to eliminate expansion loops; (2) support of heavy valves and blind flanges by close spacing of heavy concrete piers; (3) low gas velocities from 5 to 6 feet per second in the high-pressure discharge lines by use of large suction and discharge lines; (4) use of large volume suction and discharge chambers close to the compression cylinders (volumes given as cylinder volumes), 19 volumes—1500-pound suction and 4000-pound discharge, 12 volumes—450-pound suction and 1500-pound discharge; (5) use of a new type pulsation dampener on the cylinders discharging gas at 1500 and 4000 pounds with one pulsation dampener serving one to three cylinders, depending on the method of manifolding.

Residue gas from the high-pressure absorber at 1500 pounds flows through the high-pressure residue scrubber to the second stage of compression. To prevent liquid carryover from reaching the cylinders of the compressors, the residue scrubber is equipped with a mist extractor, a high-liquid-level alarm, and a high-liquid-level shutdown switch to ground the magnetos on the compressors. Each suction scrubber is equipped with this safeguard. Injection lines to the wells are equipped with check valves to prevent back flow of gas when the compressors are shut down by the safety system.

Other gases compressed are the fixed gases from the stripper reflux accumulator, hot-rich-oil vent accumulator and stripped accumulator, which are at approximately the same pressure. These gases are delivered to the compressors at 70 pounds and discharged at 190 pounds. Overhead liquids from the stripper and crude rectifier are blended with this gas for the sponge effect. Then liquid and gas flow through coolers to the depropanizer charge tank, which is also the gas separator for the second-stage recompressor. This gas is compressed from 185 to 450 pounds, then flows through a cooler and accumulator to the 450-pound high-pressure reabsorber.

One of the outstanding features in the compressor plant is the suction and discharge manifold piping which eliminated expansion loops. All the lines are supported by concrete piers spaced 6 to 8 feet apart and are fastened with three types of specially designed clamps; the type clamp used depending on the direction of pipe movement. The hold-down type clamp allows the pipe to move longitudinally. It is identical to the conventional anchor clamp with the exception that the tie-down bolts on one side are spring loaded. The box-type clamp is a rectangular clamp with the anchor bolts spring-loaded on one side. This clamp permits both longitudinal and lateral movement of the pipe and is generally used on the first two piers on either side of the corner when a line changes direction.

The suction header and the discharge header extend the full length of the compressor building. Each header is securely anchored at its midpoint and expands in both directions from this point as temperature increases. Box-type clamps are used on the remainder of the piers to permit lateral and longitudinal movement of the headers, and on the suction and discharge lines from the headers to the building.

Dual Emergency Shutdown System

Installation of a dual emergency shutdown system, primary and secondary, for the processing equipment and utilities, respectively, safeguards the plant. Control stations for both primary and secondary systems are located at five different points. The system has a maintained air pressure of 25 pounds and actuates the diaphragm- or piston-operated valves when the pressure is decreased. Each station has two bleed valves equipped with cams to insure operation of the primary system first, and then if necessary, the secondary system can be put into operation.

When the primary system is in operation, it performs four functions: (1) incoming gas to the plant, except auxiliary fuel, is shut off; (2) compressor engines and lean-oil pumps are shut down; (3) fuel

to the rich-oil heater is shut off and the firebox steam-purged; (4) pressure on the 4000-, 1500-, and 450-pound gas lines and on the 1500-pound absorber and 450-pound reabsorber is released.

The secondary system performs two operations on the utility equipment: (1) shuts off fuel to the steam boilers and steam-purges the fireboxes, and (2) shuts down the gas engines driving the electric generators.

Process

Lean oil is pumped to the 1500-pound absorber at a rate of 270 gallons per minute by three 3.5 by 12 horizontal duplex pumps. A four-cylinder, 185-horsepower, four-cycle, gas engine powers each pump. Any two of the pumps are capable of delivering the required quantity of lean absorption oil at rated engine speed. Absorption oil for the reabsorbers is pumped with two turbine-driven, six-stage, centrifugal pumps. Either of the two identical pumps can supply the 230 gallons per minute required for the two reabsorbers. Total oil circulation in the plant is about 500 gallons per minute and required makeup oil is about 80 gallons per day. About 600 gallons per day of heavy ends are picked up in the absorbers.

Rich absorption oil from the 1500-pound absorber flows through a liquid-level-controlled valve and is flashed in the high-pressure reabsorber at 450 pounds. From this reabsorber, the rich oil flows through a liquid-level-controlled valve into a horizontal vent tank. Flashed gas flows to the low-pressure reabsorber horizontal vent tank. Rich oil from the vent tank is heated from 95 to 404° F. in heat exchangers by the hot lean oil from the stripper and fractionator towers. Then the rich oil flows into the hot rich-oil flash tank operated at 135 pounds. From the flash tank, the oil is heated in the rich-oil heater to 500° F. and charged to the stripper column at 85 pounds. Hot lean oil from the stripper flows through the lean-rich-oil exchangers, which reduces the temperature from 482 to 137° F., and through the lean-oil coolers for final cooling to 80° F., then to the lean-oil surge tank. Approximately 20 minutes detention time is allowed for settling of entrained water.

A lean oil having a relatively low vapor pressure is drawn from the base of the stripper column and raw gasoline is produced overhead. The internal reflux on the first tray above the feed is drawn from the stripper, which is 5.5 feet in diameter and 52 feet high. Of the 20 trays in the tower, 8 are on 18-inch centers in the rectifying section and 12 are on 24-inch centers in the stripping section.

The side stream from the stripper, consisting principally of light absorption oil with some heavy gasoline and kerosine, is charged to the fractionator. Stripped absorption is drawn from the base of the fractionator, kerosine from a side stream connection, and natural gasoline overhead. Operation of the tower, 3 feet in diameter and 63 feet high, with 24 trays is 5 pounds. It has a 13-tray rectifying section and a 11-tray stripping section. Four trays in the stripping section are below the internal heater. Kerosine side stream connections are on the 7th, 9th, 11th, and 13th trays from the top. Elimination of light ends and vapor-pressure control of the lean oil has been possible with the design and operation of the fractionating tower.

The end-point of the obsorption oil is controlled by an oil reclaimer alongside the stripper, with a feed of 3 to 5 gallons per minute from the 13th tray from the top of the stripper. Process steam for the stripper first passes through the reclaimer, the partial pressure effect of the steam vaporizing the light ends and carrying them back to the stripper. Reclaimer bottoms are dumped periodically.

Fractionation

The crude-debutanizer tower feed is raw condensate from the 60-pound trap. The temperature of the feed is raised from 97 to 250° F. in passing through feed-to-bottom exchangers. Debutanized condensate flows from the reboiler through a heat exchanger and a cooler to storage. Overhead vapors, after condensing are pumped into the recompressor interstage discharge line ahead of the cooler. Mixed with the condensable overhead from the stripper tower, liquids and non-condensables flow to the depropanizer-charge tank.

Operating at 350 pounds, the depropanizer produces propane and lighter fractions overhead. The condensables are pumped to the injection wells by a 22 x 4.5 x 20 simplex pump which discharges at a pressure of 3750 pounds. Propane is recovered from side stream connections on the 3rd, 5th, and 8th trays from the top of the tower. Depropanized liquid from the column's reboiler flows through bottoms to feed exchangers and through the debutanizer bottoms to feed exchangers for charging to the debutanizer column operating at 150 pounds. Natural gasoline is produced from the base of the debutanizer and the butanes overhead. After condensing, the butanes are charged to the butane-splitter column operating at 115 pounds. The tower is 5.5 feet in diameter and 112 feet high with 65 trays. Isobutane is taken overhead, condensed, and the net make sub-cooled for storage. Normal butane is taken from the bottom of the tower.

One of the five emergency shutdown control stations in the Coles Levee plant. The valves are equipped with cams to insure operation of the primary system first, which shuts down the processing equipment, and the secondary system last, which shuts down the utilities. The system has a maintained air pressure of 25 pounds and operates shutdown valves when pressure is decreased.

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EXPERIENCE has proved that in a majority of installations Revere Admiralty Metal condenser tubes, inhibited to resist dezincification, will give long, satisfactory and economical service. However, Revere also makes tubes in Cupro-Nickel, Revalon (aluminum brass), Red Brass, Muntz Metal, Arsenical Copper, and Copper, thus enabling you to select the Revere Metal or Alloy best suited to meet your operating conditions.

In addition, Revere manufactures pipe and tube in copper and brass for general piping purposes, and electric welded steel tube.

Revere condenser plates can be had in a number of alloys in squares, rectangles, circles, half-circles, segments, and special patterns, up to 120 inches maximum dimension, finished weight about 11,000 lbs. maximum. These plates are flat, dense, free from blow holes or other imperfections, accurate in gauge, and are easily machinable.

As each condenser, heat exchanger or other piece of equipment using tubes and plates presents an individual combination of conditions, no blanket recommendations are possible. Among the factors to be considered are: chemical and impingement attack, velocities, pressures, temperatures, air entrainment, suspended solids. Should short tube life or other circumstances lead you to question your present practices, the Revere Technical Advisory Service will gladly cooperate with you in making thorough field and laboratory studies, to assist you to determine the most serviceable and economical alloys.

REVERE

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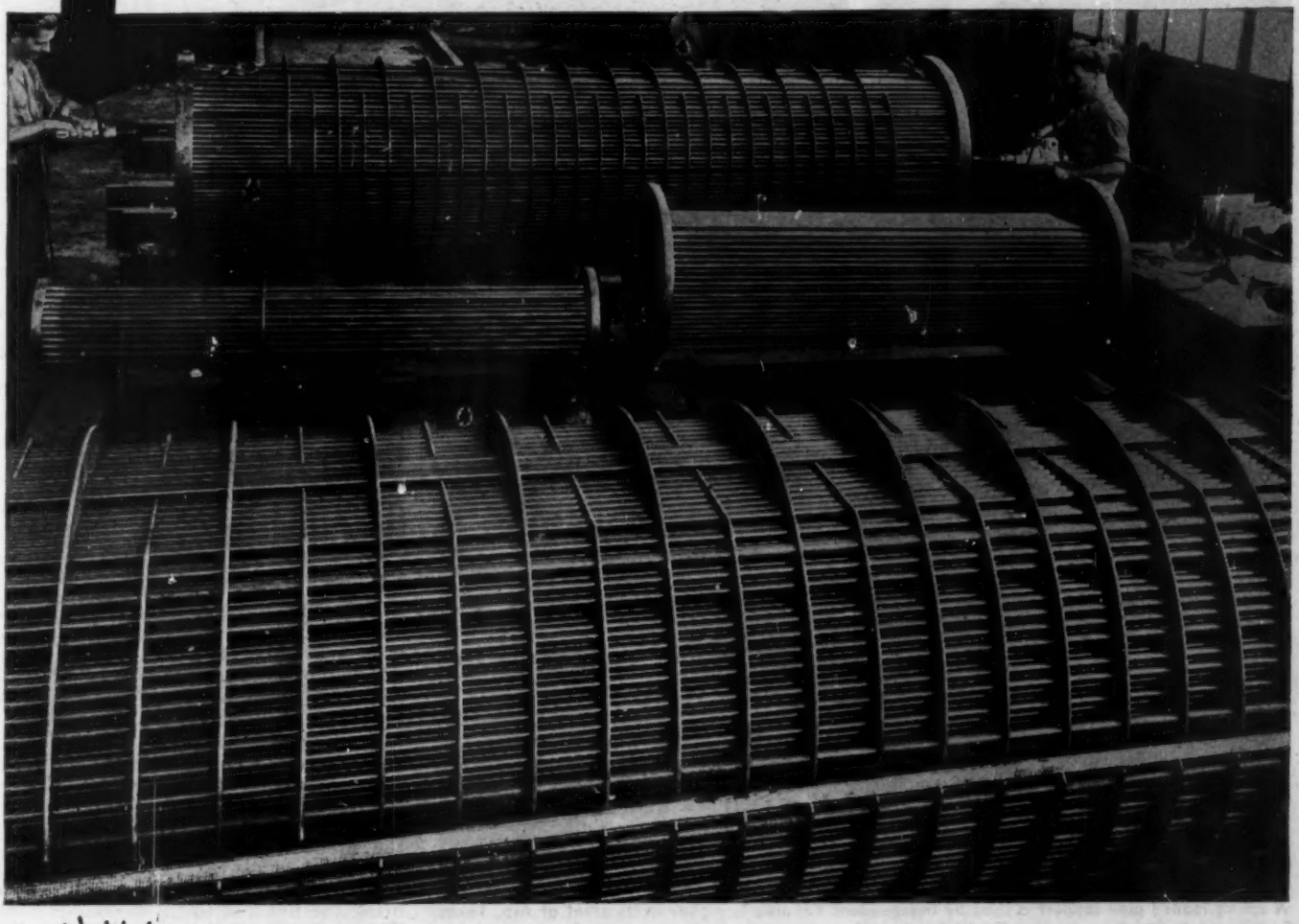

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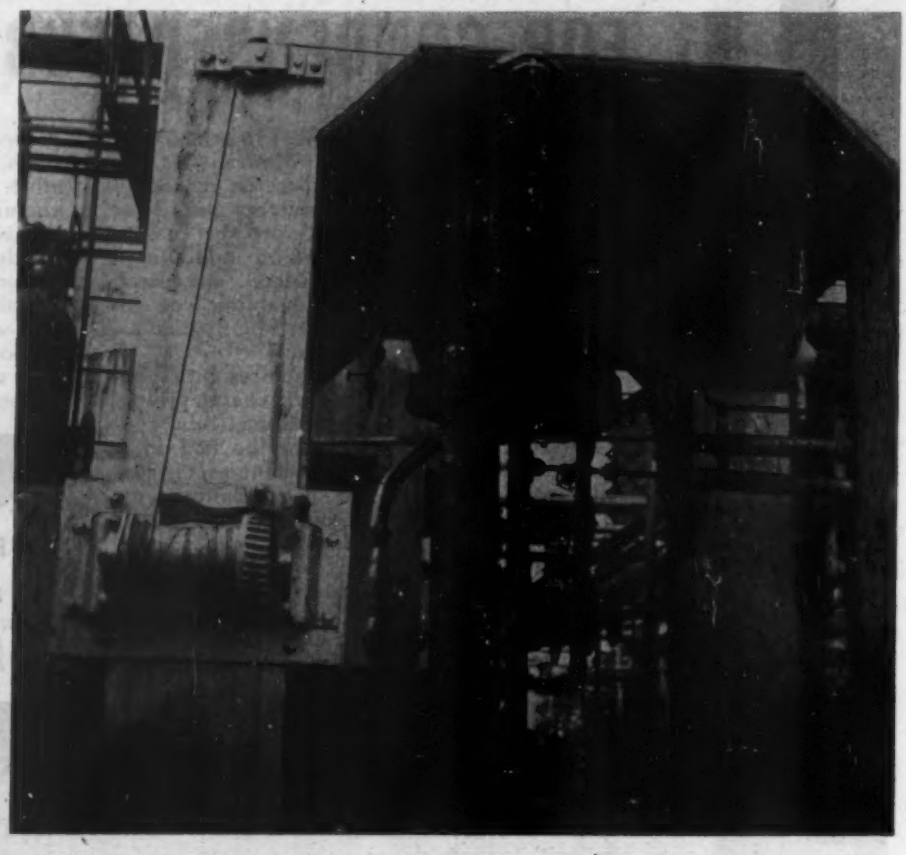
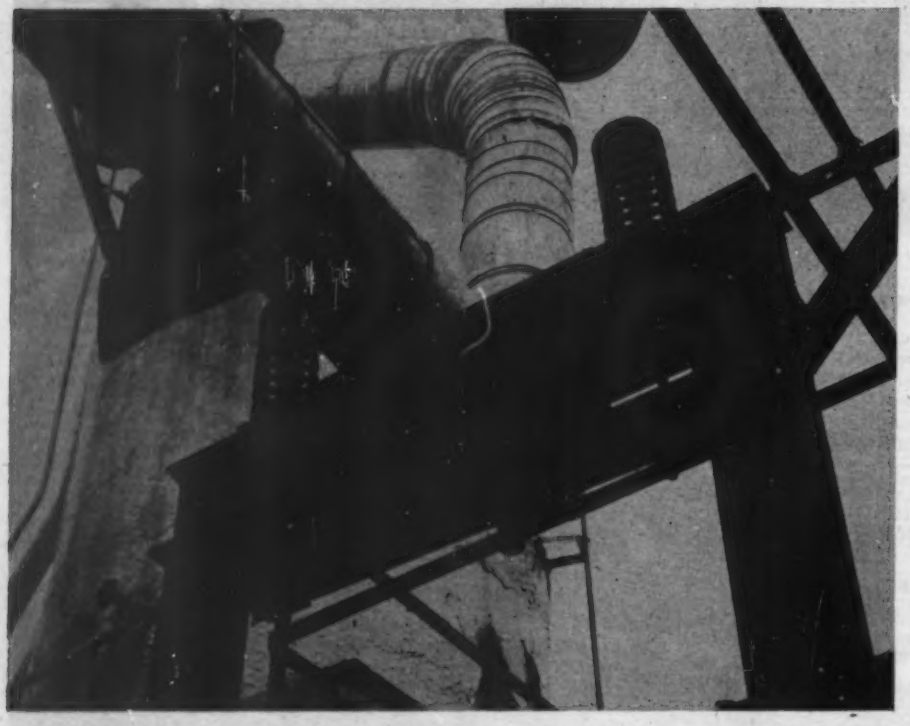
OPERATION HOW TO DO IT MAINTENANCE

Hoist Solves Problem of Heavy Manhole Cover

MANHOLE covers located on the bottom head of high-pressure refinery vessels are usually so heavy that they are difficult to handle when they must be removed for cleanout or inspection purposes. The Independent Refining Company at Arp, Texas, has installed a cable, pulley, and hoist arrangement to meet such a problem on one of the process vessels. The manhole cover is provided with hinges and the load involved is such that one man can raise the cover into the exact position required for bolting, or can hold the weight of the cover to allow easy removal of bolts when the manhole is to be opened.



Hoist and pulley arrangement for handling heavy cover of manhole at bottom of process vessel. Connection at manhole is shown in smaller picture.



A spring loaded pipe support is used by Independent Refining Company in its plant at Arp, Texas. Springs are built into a structural-steel frame under a furnace transfer line in such a manner that thermal expansion of the reaction vessel fed by the transfer line is compensated and reasonably constant support is provided to the line.

Boiler-Blowdown Valve Operated By Extension

WHERE an oilfield (locomotive) type boiler is used to furnish process steam, it is necessary to provide for clearing the unit of mud and sludge at intervals which depend upon the water used and its degree of hardness.

In one plant where this type of boiler is used the working valve is offset from the center line of the boiler, so as to afford direct shaft operation of the stem from the fireman's position near the firedoor. The usual block valve is placed as close to the boiler shell as possible, with a flange joint and ell to bring the line out to the working valve. This unit is supported on the boiler foundation with a clamp which takes the weight but which does not cramp shift-

PENBERTHY

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DROP FORGED STEEL LIQUID LEVEL GAGES

Liquid always shows black—empty space shows white. Preferred where the liquid level must be easily and positively visible, and when liquids are under high pressure or at high temperature. Made of alloy temperature resisting steel and are highest quality throughout. Liquid chamber made from solid block of steel to assure perfect alignment and rigidity. Conform with A.P.I.—A.S.M.E. requirements.

The "Reflex" is one of the complete line of Penberthy Gages that meet every liquid level gage requirement.

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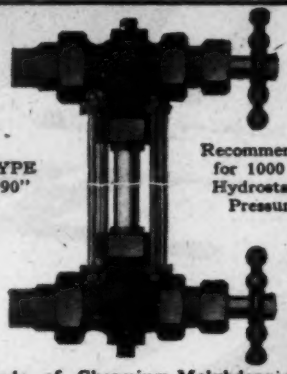
DROP FORGED STEEL

LIQUID LEVEL GAGES



TYPE "90"

Recommended for 1000 lb Hydrostatic Pressure

Made of Chromium-Molybdenuin alloy temperature resisting steel, extra heavy throughout and stainless steel trimmed. Positive automatic shut-off; stainless steel balls shut off the flow of liquid if glass breaks. Regrinding removable seats, heavy duty stuffing boxes, union connections. Conform with A.P.I.—A.S.M.E. requirements.

This is one of the complete line of Penberthy gages that meet every liquid level gage requirement.

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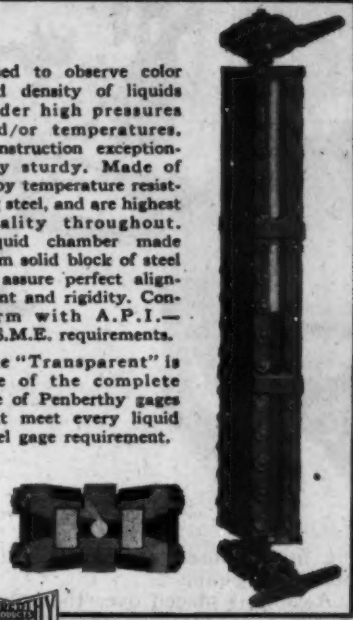
PENBERTHY

"TRANSPARENT"

DROP FORGED STEEL LIQUID LEVEL GAGES

Used to observe color and density of liquids under high pressures and/or temperatures. Construction exceptionally sturdy. Made of alloy temperature resisting steel, and are highest quality throughout. Liquid chamber made from solid block of steel to assure perfect alignment and rigidity. Conform with A.P.I.—A.S.M.E. requirements.

The "Transparent" is one of the complete line of Penberthy gages that meet every liquid level gage requirement.



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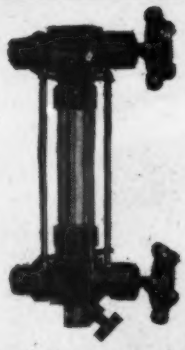
DETROIT, MICH. Canadian Plant WINDSOR, ONTARIO

PENBERTHY

"ALL IRON"

LIQUID LEVEL GAGES



Body made from a special high strength alloy iron, shanks alloy steel and trim stainless steel. Extra heavy construction throughout, automatic and positive shut-off if glass breaks, heavy duty stuffing boxes. Conform with A.P.I.—A.S.M.E. requirements.

The "All Iron" is one of the complete line of Penberthy gages that meet every liquid level gage requirement.

PENBERTHY INJECTOR CO.

DETROIT, MICH. Canadian Plant WINDSOR, ONTARIO

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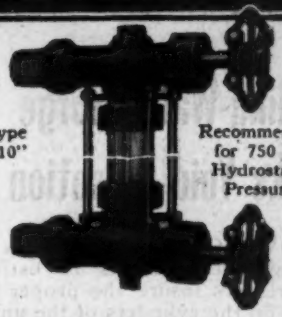
DETROIT 2, MICH. Canadian Plant WINDSOR, ONTARIO

PENBERTHY

DROP FORGED STEEL

LIQUID LEVEL GAGES



Type "10"

Recommended for 750 lb. Hydrostatic Pressure

Steel construction throughout with stainless steel interior trim. Automatic shut-off is positive and instantaneous; stainless steel balls shut off the flow of liquid when glass breaks. Heavy duty stuffing boxes, union connections, offset construction facilitate cleaning and simplify glass replacement. Conform with A.P.I.—A.S.M.E. requirements.

This is one of the complete line of Penberthy gages that meet every liquid level gage requirement.

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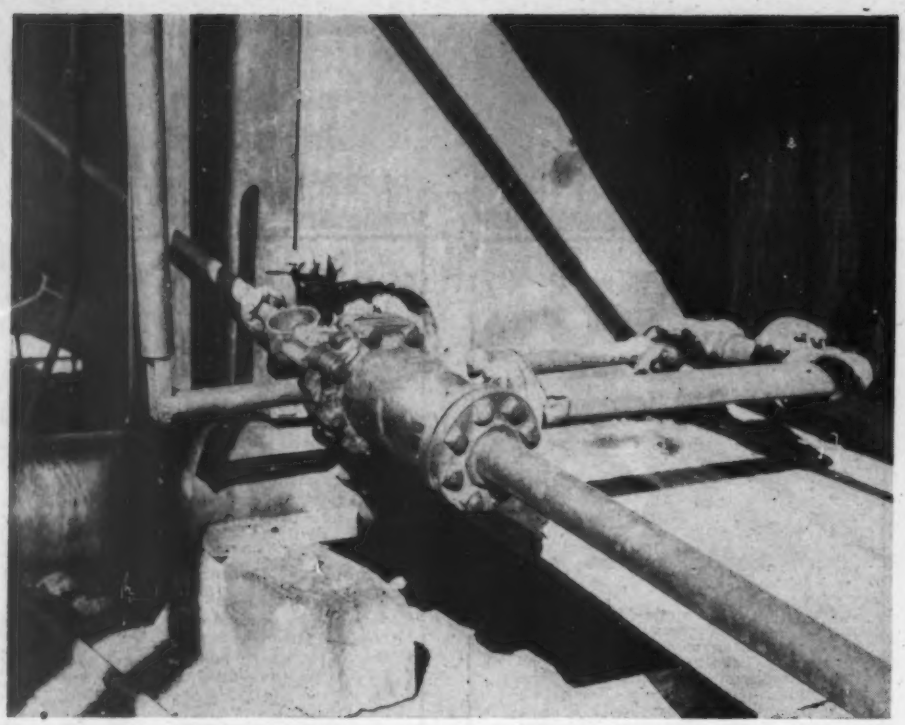
DETROIT, MICH. Canadian Plant WINDSOR, ONTARIO



Boiler-blowdown valve operated from in front of firebox.

ing in response to expansion of either boiler or piping.

A yoke is placed over the valve stem in place of the customary wheel, the outer end of the yoke carrying an automotive type universal joint. An extension shaft, its outboard end supported in a bracket at the front of the boiler, transmits torque from the handwheel there to the universal joint and thus enables the valve to be spun open or shut while the fireman keeps an eye on the water level and steam gauge.

The same bracket that supports the blow-down line also carries the extension handle for the block valve, this unit being set so as to swing through an operating arc outside the working valve. A pipe handle slipped over the crank gives added leverage and affords a cooler grip.

Cooling-Water Surge Tanks Void Pulsation

PROVISION of surge tanks on the cooling-water systems to batteries of compressors insure the proper head of water on the cylinders of the units at all times, and eliminates the pulsations which sometimes are set up when delivery of water is direct from cooling-tower sump to the compressors. The surge tanks also serve to separate out from the large volumes of water handled any air which may have been picked up by the steam on its way through the tower.

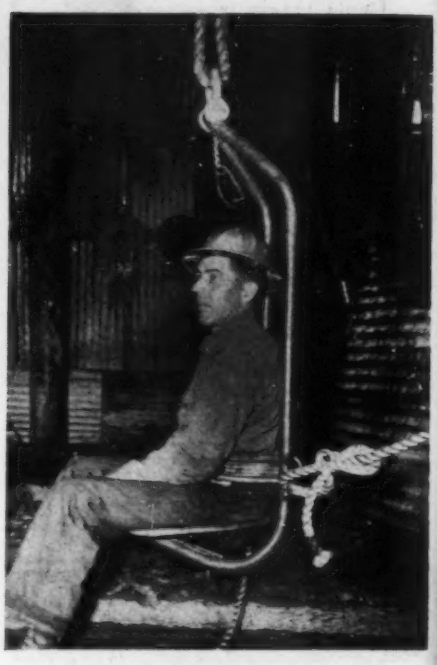
Since the optimum working temperatures of compressor cylinders and power cylinders vary by some 20 degrees, it is customary to employ separate sections of the tower for the two circuits, and to equip each circuit with its own surge tank.

Volume of the surge tanks is sufficient to insure full head of water to the compressors long enough to enable them to be shut down without danger of injury from overheating in case of breakdown of the system circulating the cooling water over the tower.

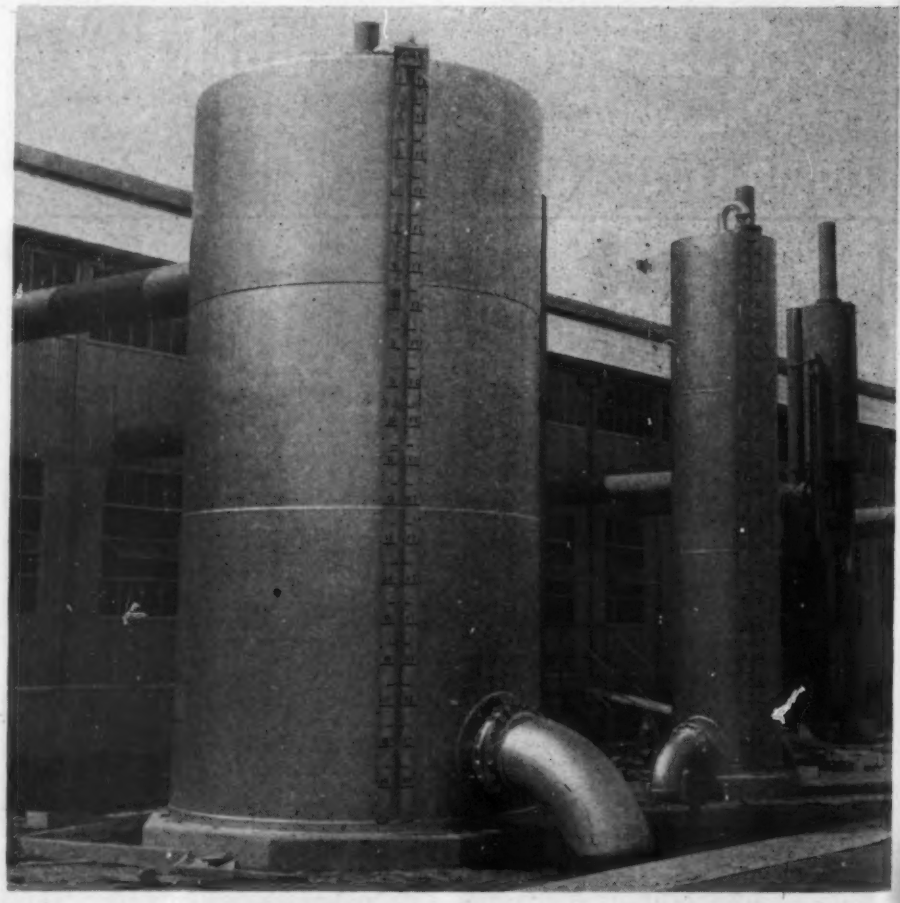
Gauge boards outside each surge tank indicate the head available on each of the cooling systems and provide visual check against indicating units on each of the compressors.

Steel Chair for Safe Working Above Ground

THE usual "bo's'n's" chair, in which the occupant sits on a narrow board between the two ropes of the bight supporting him, does not allow the freedom of movement which is sometimes re-

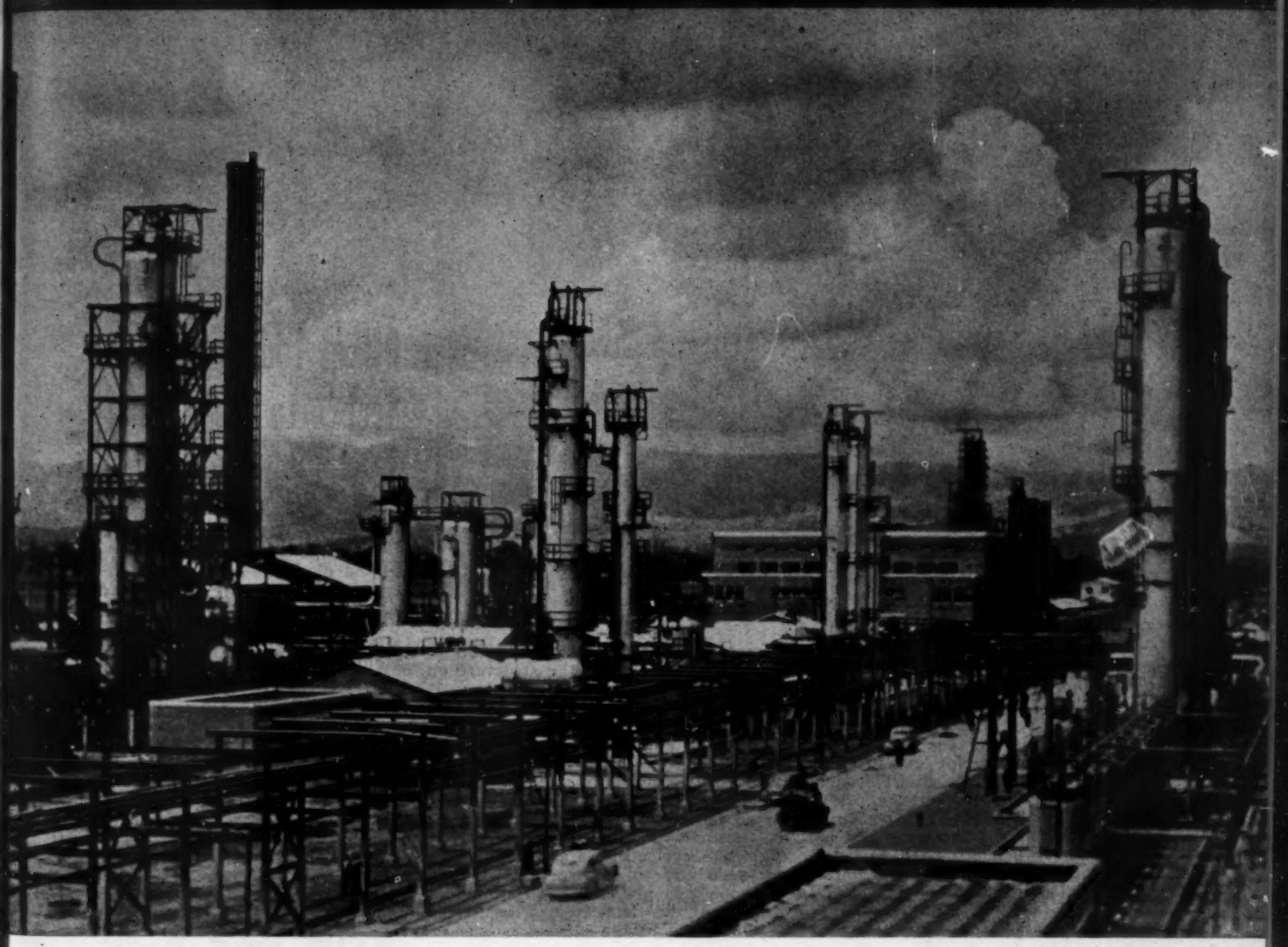


Steel chair for "high-up" work.



Cooling-water surge tanks at compressor station.

UNDIVIDED RESPONSIBILITY IN ONE ORGANIZATION



UNDER a single contract, Arthur G. McKee & Company are designing and building the following projects for Petroleos Mexicanos:

40,000 barrel refinery at Atzcapotzalco.

65,000 barrel crude stabilization plant and 75,000,000 cubic foot absorption plant at Poza Rica.

Doubling of crude oil pumping facilities from Poza Rica to Mexico City.

This is an example of the McKee method of Undivided Responsibility in one Organization.

Arthur G. McKee & Company

★ *Engineers and Contractors* ★

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Offset ladder on elevated tank structure.

quired for working on the outside of some of the high structures now in use in many refineries.

One company uses an all-steel welded chair which gives the operator full clearance in front and to both sides. The frame consists of a pipe unit, formed to support a steel cultivator seat at the base, and to carry a clevis at the top for attaching to the hoisting rope or wire.

At the proper height above the seat is a webbed safety belt, attached to both of the pipe uprights through loops which permit lateral shifting. At the right of the seat is a 4-foot length of chain with harness snap on the free end, which the operator may use to attach the unit to any convenient part of the structure on which he is working. A second short chain, suspended below the clevis, forms a point for attaching a spotlight if concentrated illumination is required for a night job.

Each of the uprights carries a loop at safety-belt level through which may be passed the end of a guide rope, so that an assistant at the ground level may guide the service man's ascent and help him keep clear of obstructions. Use of this rope also prevents the spinning or whirling of the chair as it is hoisted by the line through the clevis.

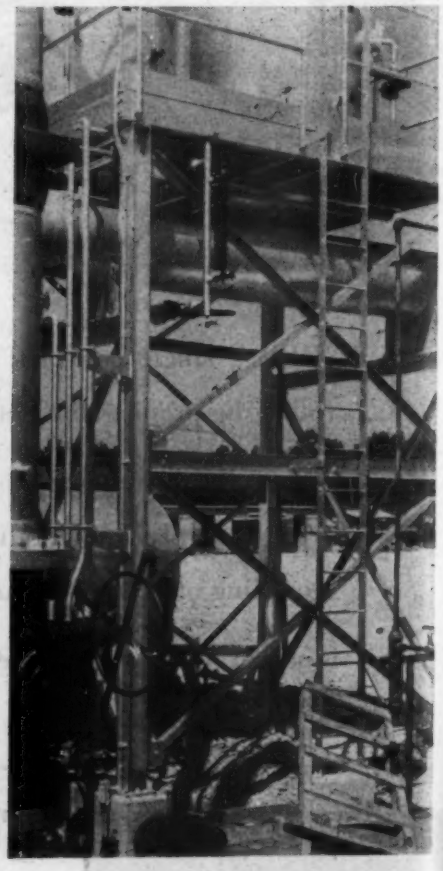
Assemblies consisting of a lubricator, strainer, plug cock and hose couplings, bolted to furnace handrails in convenient locations for use with air tools during furnace cleanout periods have proven a time saver in the plant of the Independent Refining Company at Arp, Texas.

Damper Device Works Through Universal Joint

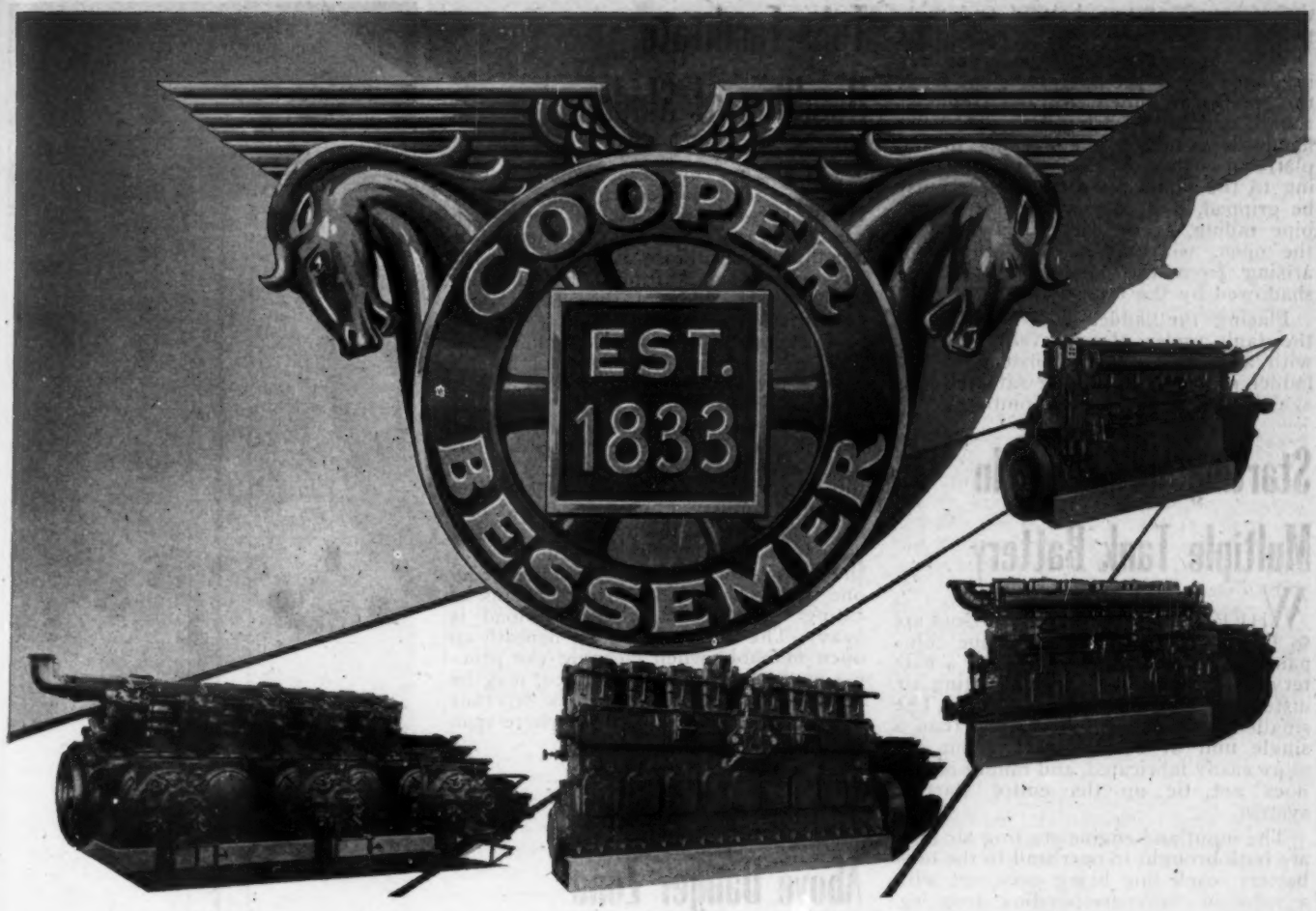
DAMPERS on refinery furnaces require precise positioning and should be capable of this adjustment with a minimum of time and effort on the part of the operators. Skelly Oil Company in its refinery at Longview, Texas, has constructed a damper-control device which has been very satisfactory. The device consists of a notched angle ring with a rotating lever and spring catch which is connected to the furnace damper with shafting and a universal joint. The damper can be positioned at any desired angle and will then stay in place, held by the spring catch which engages the notched angle ring.

Offset Ladder for Safe Access to High Tanks

LADDERS to elevated tanks are usually run up flush with the edge of the platform on which the tank is set, making it necessary for the operator to feel for the top step under the overhang of the platform—a condition which introduces an unnecessary hazard. One plant, using the customary welded ladder with rounds set into 1½ x ½-inch steel risers, located the ladder some eight inches from the platform, supporting it there with brackets attached to



Device for precise positioning of furnace damper.



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the structure at top, bottom and midpoints.

With the ladder in this position, it is in plain view as the operator steps from the platform down to the topmost round; without releasing his grip on the handrails enclosing the opening to the platform enclosure. Then, after descending to the point where the risers must be gripped, it is easy to shift from the pipe railing, as the change is made in the open, with no possible confusion arising from the ladder being overshadowed by the tank unit.

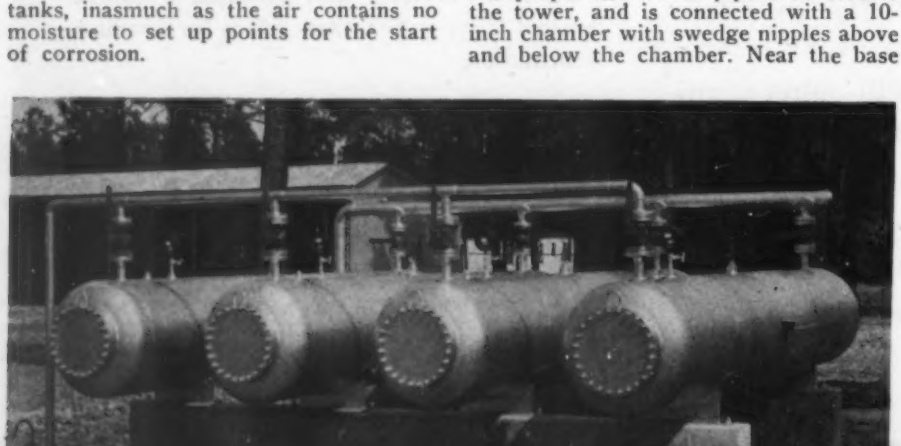
Placing the ladder well away from the tank tower also prevents contact with any of the braces instead of the ladder rounds, still further safeguarding against accidents at this point.

Starting Air Stored In Multiple Tank Battery

WHERE a number of compressors are to be air-started at intervals, one company finds it advantageous to use a battery of tanks for storing the starting air instead of relying on one larger unit. The smaller tanks require more steel than a single unit of equal capacity, but are more easily fabricated, and failure of one does not tie up the entire starting system.

The input and engine-starting air lines are both brought in overhead to the tank battery, each line being equipped with a valve in the corresponding drop leg. The four tanks may all be placed on either line, or may be collectively interconnected so that all compressed air enters one tank, passing through it and thence to the other tanks through what normally are discharge valves—the inlet valves being closed on the other three units.

This system makes one tank in effect a condensation trap for the elimination of any moisture which may be trapped in the system, insuring dry air in the other three tanks. The receiving tank may then be closed off and blown dry without affecting the pleasure of the other three. It has been found that this system of diverting all incoming air through one tank practically eliminates internal corrosion of the other three tanks, inasmuch as the air contains no moisture to set up points for the start of corrosion.



Starting-air storage tanks.

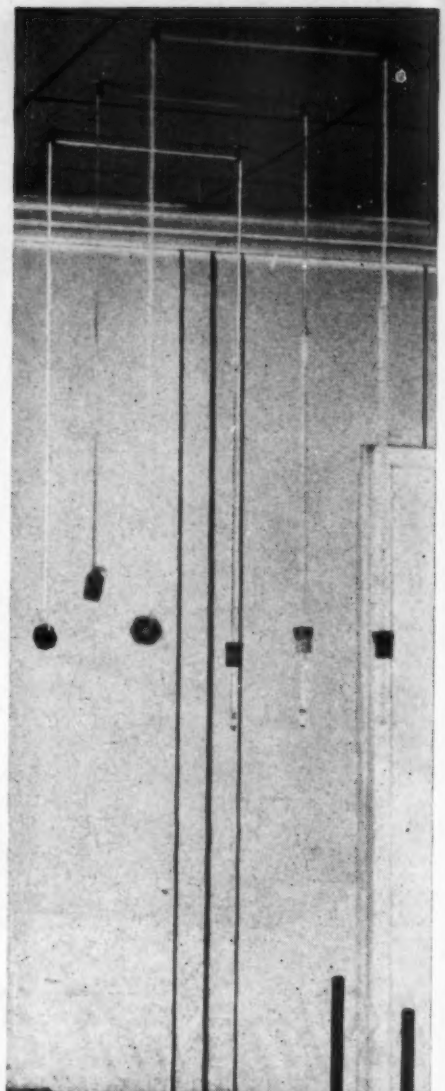
Tubs Facilitate Handling of Sludge

FOR moving considerable quantities of sludge, or discarded treating clays, wheel barrows and dump trucks are sometimes not sufficient. One method of handling such refuse is to do it with large tubs, having lifting bails and dump hooks on the side of the vessel. Any cylindrical vessel may be used, such as rings from columns relegated to the junk pile, or even the waist of boilers can be pressed into service if available. If no floor, or bottom is in the section of the cylinder, tank steel cut circular is welded to form the bottom. For handling, eyes are welded to the rim of the tub into which the bail is installed. The bail itself must of necessity be strong and rugged to lift the loads required and survive rough handling. A hook near the bottom on one side, 90 degrees from one of the bail eyes provides means of tilting with power when the load is heavy. The tubs may be set beneath an open manhole when cleaning the process equipment, and when filled, may be lifted with a boom on a truck or crane and transported to the point where such refuse is dumped.

Vapor Vent Elevated Above Danger Zone

VAPORS vented from gasoline and light naphtha tanks, either intentionally or accidentally, normally are conducted to a flare, but in The Newhall Refining Company plant at Newhall, California, due to erratic winds surging through the mountains and canyons, the vent line is built alongside the main fractionating column, reaching several feet above the top. In this manner all inflammable vapors are dissipated before they can settle to the ground.

Not only is the vent line designed for disposing of vapors, but is built upon, and is included with a liquid disposal system which diverts inflammable liquids from the area into slop tanks. The vent line proper is a 4-inch pipe bracketed to the tower, and is connected with a 10-inch chamber with swedge nipples above and below the chamber. Near the base of this chamber is a bolted manhole for inspection. Inside the chamber is a floating ball, resting upon a seat at the base when the vent is inactive, but raised by liquid if and when a surge tank is filled above its capacity. When liquids above the capacity of the disposal line leading from the base of the chamber to the slop tank enters the system, the floating ball seats in the upper part of the chamber, sealing off both vapors and liquids until they are forced to travel to the slop tank. When the liquid level falls to remove the floating ball from the upper seat, vapors again flow upward through the vent line to the top of the tower.



Skelly Oil Company in the laboratory of its Longview, Texas, refinery has installed the thermometer counterweights shown in the photograph over a battery of ASTM distillation test apparatus. When a thermometer is in use it is pulled down and inserted into the neck of the distillation test flask. When the test is complete the thermometer is removed and allowed to wait for the next test run. It is believed that thermometer breakage has been lessened by this gadgetry, and it is certain that a little of the tester's time is saved.

Liquefied Petroleum Gas In 1946

G. G. OBERFELL, Vice President in Charge of Research and Development,
Phillips Petroleum Company

R. W. THOMAS, Manager Research and Development,
Phillips Petroleum Company

SUMMARY

THIS memorandum discusses the growth and development of the liquefied petroleum gas industry for the year 1946. It contains a review of volume of marketed production beginning with the year 1922 through 1945, and presents estimates of the volume of marketed production for 1946.

It is estimated that total sales for 1946 increased 33.5 percent over 1945, to a total of 1,425,000,000 gallons. The greatest increases in the use of liquefied petroleum gas during 1946 were experienced in the distribution for household purposes, gas manufacturing and for chemical manufacture.

ANYONE studying the growth of the liquefied petroleum gas industry will at once recognize the tremendous strides made in volume of product marketed. Particularly is this growth impressive when one considers that as of the end of 1946 the annual marketed volume is thirteen times the volume that it was ten years ago. Not only has the increase in the marketed volume of these products approached staggering proportions but in addition the methods of handling, selling and utilizing these products have developed to a stage of refinement and acceptance that liquefied petroleum gas now is regarded as a necessary and dependable fuel equal to or better than the best available materials for similar use. The past year is the first full year since mid 1941 in which the industry has operated without direct wartime restrictions. The development and growth of the industry during 1946 would have reached even greater proportions had it not been so unduly restricted by the same material and man-power shortages as were experienced by most of the other industries in the country during this readjustment period.

It is worthy of particular note that the total estimated marketed production of LP-gas during 1946 amounted to a little more than three times the marketed production of LP-gas in 1941, the last full year of production and sales prior to the war. It is estimated that the total marketed production of LP-gas in 1946 (1,425,000,000 gallons) exceeded the marketed production of 1945 by 33.5 percent. Particularly for household use and for gas manufacturing the demand far exceeded the marketed volume which was limited by the unavailability of utilization and transportation equipment.

Domestic Use

It is estimated that at the close of 1946 there were approximately 3,500,000 homes using liquefied petroleum gas for household purposes. In addition to these household uses, this particular classification of marketed production (See "Domestic" in the table) includes the many other requirements for which these customers desired LP-gas, such as irrigation pumping, tractor fuel, chicken brooding and the many other demands that existed for a fuel of this nature. Included in this classification of marketed production there is a considerable volume which was used for industrial purposes, internal-combustion engines and for gas-manufacturing. This consumption was tabulated in this classification because it was sold by producers to distributors whose principal sales were for household purposes.

In spite of the shortage of steel for tanks, cylinders and appliances which limited the number of new installations, the increased use of the gas on the part of continuing customers and the demand on the part of new customers installed during the year brought the total estimated "domestic" consumption to 763,000,000 gallons, an increase of 43.1 percent over 1945. This large increase was possible only because of the initiative and ingenuity displayed by the marketers. One evidence of this was the development of distribution methods by which a far greater number of customers was served with a given number of cylinders than was thought possible a few years ago.

Manufacturers are recognizing the tremendous demand for LP-gas-consuming appliances and their increased interest in this market is worthy of note. Another interesting trend is the increased use of meters for measuring household consumption.

The past year has seen the greatest demand for new bulk stations, both on the part of companies already in the business as well as by newcomers. This has resulted in making the distribution of LP-gas to the consumer more economical because of shorter hauls, a factor that brings lower cost to the operator and an advantage in obtaining greater consumer saturation. Indications are that this trend will continue. It is estimated that approximately 2500 bulk distributing plants are in operation now.

Industrial Use

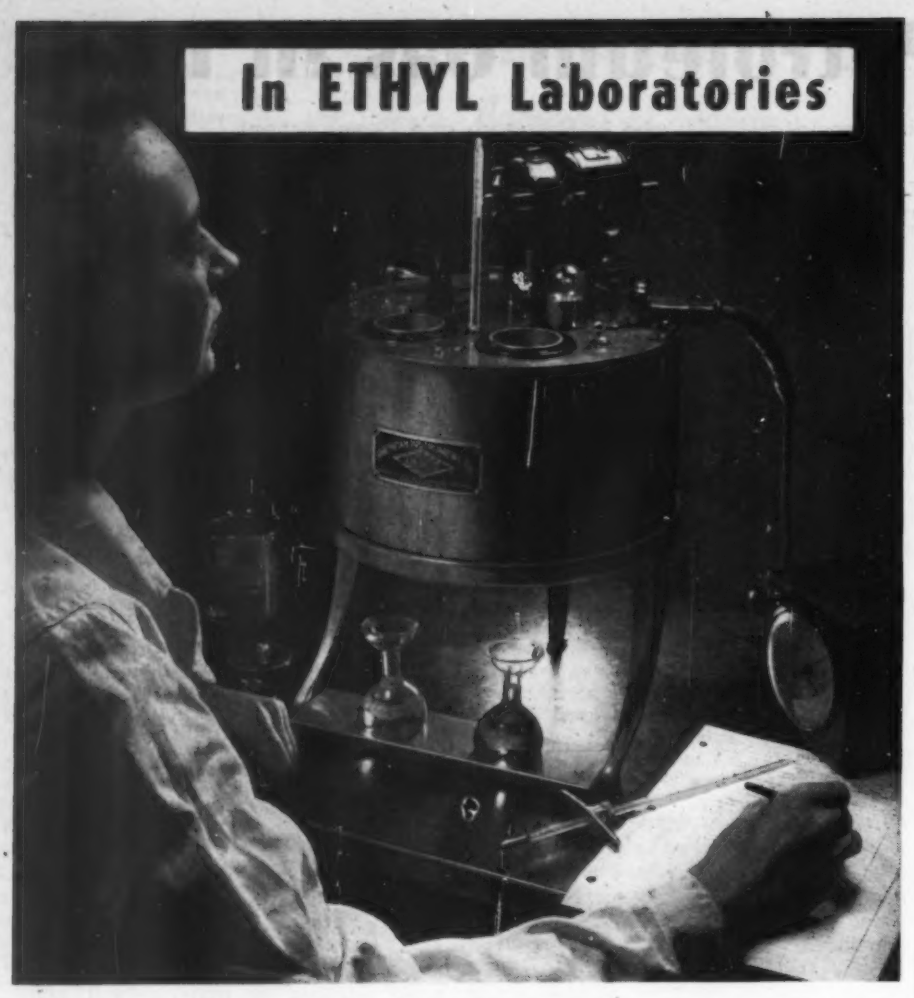
A number of industries were greatly curtailed or completely shut down in the conversion from wartime to peacetime activities and very little growth in LP-gas volumes consumed for industrial use was experienced during the past year. Because of material and manpower shortages an increased demand in existing industrial installations and

Marketed Production of Liquefied Petroleum Gas
(In Thousands of Gallons)

YEAR	TOTAL SALES		DISTRIBUTION							
	Gallons	Percent Increase	Domestic	Percent Increase	Industrial and Miscl.	Percent Increase	Gas Manufacture	Percent Increase	Chemical Manufacture	Percent Increase
1922	223	.....								
1923	227	24.4								
1924	376	36.0	Sale of Liquid Petroleum Gas Confined Primarily to Bottled Business Prior to 1928.							
1925	404	7.2								
1926	465	15.2								
1927	1,091	134.6								
1928	4,523	314.6	2,600	.....	400	.....	1,500	.....	.....	.....
1929	9,931	119.6	5,900	126.9	1,500	275.0	2,500	66.7	.....	.....
1930	18,017	81.4	11,800	100.0	2,200	46.7	4,000	60.0	.....	.....
1931	28,770	59.7	15,295	29.6	7,172	226.0	6,303	57.6	.....	.....
1932	34,115	18.6	16,244	6.2	8,167	13.9	9,703	53.9	.....	.....
1933	38,931	14.1	16,626	2.3	13,987	71.3	8,318	—14.3	.....	.....
1934	56,427	44.9	17,681	6.3	32,448	132.0	6,298	—24.3	.....	.....
1935	76,855	36.2	21,380	20.9	47,894	47.6	7,581	20.4	.....	.....
1936	106,652	38.8	30,014	40.4	67,267	40.4	9,371	23.6	.....	.....
1937	141,400	32.6	40,823	36.0	62,610	*	11,175	19.3	26,792	.....
1938	165,201	16.8	57,832	41.7	62,694	0.0	12,386	10.8	32,299	20.5
1939	223,580	35.3	87,530	51.4	93,723	49.4	15,435	24.6	26,892	—16.7
1940	313,456	40.2	134,018	53.1	124,482	34.5	20,285	31.4	34,671	29.0
1941	462,852	47.7	220,722	64.7	172,669	38.6	25,255	24.5	44,206	27.5
1942	585,440	26.5	299,559	35.7	201,447	16.7	31,366	24.2	53,038	20.0
1943	675,233	15.3	339,380	13.3	242,978	20.6	37,519	19.6	55,356	4.4
1944	898,071	33.0	445,617	31.4	254,590	4.8	45,879	22.3	151,985	175.0
1945	1,067,979	19.0	533,262	19.7	256,577	0.8	53,849	17.4	224,291	47.5
1946	1,425,000	33.5	763,000	43.1	257,000	0.2	80,000	48.6	325,000	44.8

* Not comparable due to segregation of chemical manufacturing.

REMARKS: In this table "Total Sales" for all years except 1946, were obtained from U. S. Bureau of Mines reports. "Distribution" for the years 1931 to 1945, inclusive, was obtained from the same source. All other volumes were estimated by the writers. The total sales volume includes all liquefied petroleum gases (propane, butane, and propane-butane mixtures) when sold as such. Until 1944 the sale of pentane when sold for any purpose other than motor-fuel blending was included. Since then it has been excluded. It does not include butane when blended with heavier petroleum fractions for motor-fuel purposes. Intercompany sales transactions such as purchases of liquefied petroleum gases by one company from other companies and resold as liquefied petroleum gases have been eliminated in order to avoid duplication of sales figures. The data do not reflect sales of liquefied petroleum gases used directly by the producer at the point of production, for fuel, polymerization, solvent de-waxing, etc. Neither do the figures include sales of hydrocarbons to plants manufacturing synthetic rubber or aviation gasoline or their components.



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in new installations did not materialize. In 1946 it is estimated that 257,000,000 gallons were used. This represents an increase of 0.2 percent over that consumed in 1945.

In addition to industrial uses, LP-gas sold directly by producers for fueling internal-combustion engines is included in this tabulation. A certain growth in this use was evident but much of the product so used was sold by resellers and included in the domestic classification.

With increased distribution of natural gas it is to be expected that some LP-gas will be replaced with this fuel. In a number of conversions natural gas will be purchased under high-cost peak-load prices or with shutoff provisions. In such cases LP-gas installations will be used for standby. As industrial reconversion progresses and manufacturers build many of their plants which are now in the planning stage, sales for industrial uses will again expand.

Use By Utilities

The largest percentage increase in consumption for LP-gas during the past year was by utility companies for gas manufacturing. The estimated sales volume was 80,000,000 gallons, an increase of 48.6 percent over 1945. Many gas-manufacturing plants operated throughout the war years in a state of low efficiency due to inability to secure replacement parts or new equipment. A number of these plants have been converted to LP-gas during the past year with an appreciable economic advantage. There are now more than 300 towns served by gas-manufacturing plants depending upon liquefied petroleum gas for their entire requirements.

An increasing number of homes were converted to natural or manufactured gas for house heating. As a result utilities are experiencing large winter peaks in the demand for house heating. The additional house-heating load has taxed their manufacturing facilities or transmission lines beyond their physical limits, resulting in the need for additional gas-producing and transmission facilities. To satisfy this peak-load requirement many utilities are installing LP-gas storage to supply this periodic deficiency in gas supply. Since the utilities' peak demand occurs at approximately the same time as does that of the LP-gas industry, it is necessary for the utilities to install sufficient LP-gas storage to satisfy this seasonal demand. Due to the shortage of steel for manufacturing storage tanks only a portion of these projected installations were completed in 1946. Part of this huge peak load demand may be temporary, pending construction of additional gas-manufacturing facilities or additional natural-gas pipe lines; however a large portion of this demand can be considered permanent.

Chemical Uses

Liquefied petroleum gases make excellent feed stocks for the production of certain intermediate materials in the manufacture of chemicals. As a result of the impetus given by the war activity in synthesizing a great number of chemicals, this industry has continued to grow since the termination of hostilitiles.

Assuming that the demands of the same general type of chemical manufacturing activity are included in this classification as in previous tabulations, it is expected that during 1946 approximately 325,000,000 gallons of LP-gas will

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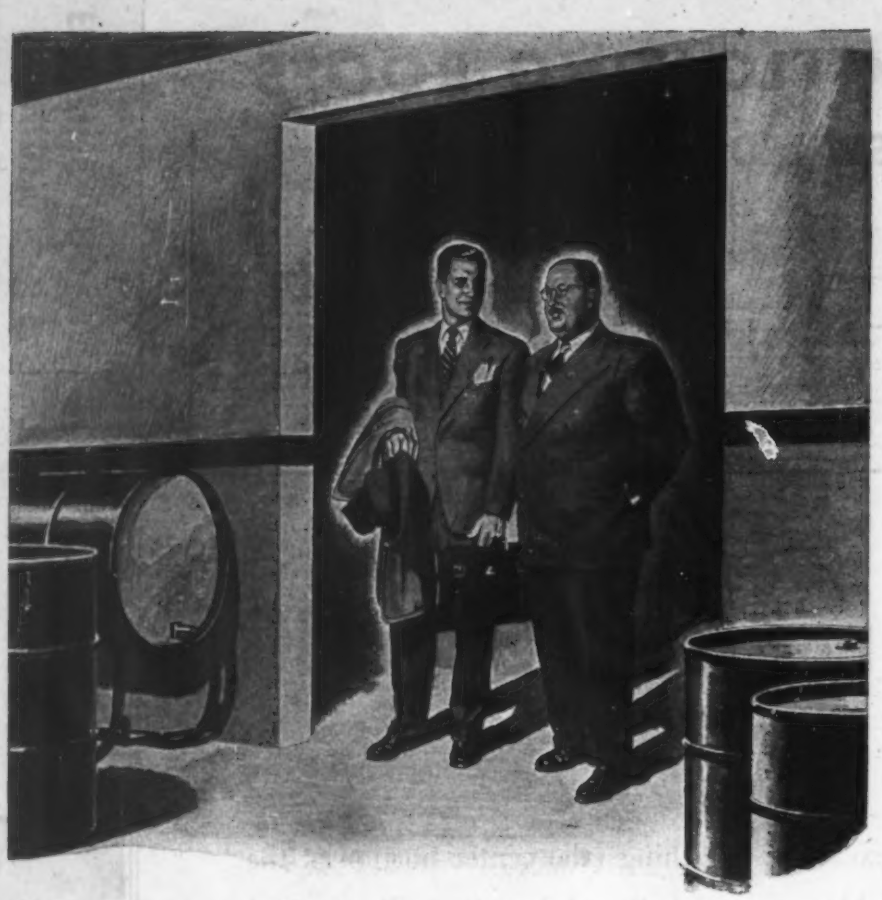

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FIRE PROTECTION HEADQUARTERS

have been consumed. This represents an increase over 1945 of 44.8 percent. The above consumption excludes the C_3 and C_4 hydrocarbons used for the production of motor fuel, aviation gasoline, and synthetic-rubber components.

Large quantities of LP-gas are still being used in the manufacture of synthetic rubber components. The plants employing raw materials derived from petroleum are still in operation and in general have not curtailed their ouptut, but rather have endeavored to produce the maximum quantity possible. Therefore the demand for this purpose has been high through 1946 and probably will continue in approximately the same amount through 1947.

Industry Activities

Accompanying the growth of the LP-gas industry has been the development of high safety standards. This activity has been advanced by national and state associations comprised of industry members, as well as by the organizations of the individual companies. One of the foremost accomplishments in establishing minimum safety requirements was the revision of the National Board of Fire Underwriters Pamphlet No. 58 entitled "Standards for the Design, Installation and Construction of Containers and Pertinent Equipment for the Storage and Handling of Liquefied Petroleum Gases as Recommended by the National Fire Protection Association." The Liquefied Petroleum Gas Association is constantly active in developing these standards. Many states and municipalities have adopted these standards as the basis for statutes and regulations to govern the conduct of the industry's activities.

An effective publicity campaign has been continued during the year by the Liqeufied Petroleum Gas Association. Electricity continues to strive for a leading role as competitor to LP-gas for the household uses.

Supply

During the war large quantities of liquefied petroleum gases were recovered in refineries for use in the production of aviation gasoline and synthetic-rubber components. Since the cessation of hostilities a large portion of the facilities producing aviation-gasoline components have been employed for other purposes. A very large quantity of the butane (and butylenes) formerly used for producing war materials is now going into motor fuel, however a good portion of it has been made available for use by the LP-gas industry. It appears that butane will continue to command a higher price than propane due to the demand for butane in motor fuel.

From available information it appears that most of the increased volume of LP-gas marketed in 1946 over 1945 was obtained from refinery sources. Large additional quantities of LP-gas are potentially available both from natural gas sources and from refinery sources. With firmer prices for propane in the future it will be possible for producers to justify its diversion from other end uses and to invest in recovery and purification equipment to bring additional product to commercial specifications.

One of the greatest shortages encountered during the past year has been in transportation facilities. Because of limitations imposed during the war on the construction of new LP-gas tank cars, the industry's fleet was taxed to capacity at the beginning of the year. The

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Maintenance engineers have long recognized Red Lead as the "standard" metal protective paint. This acceptance is based, to a great extent, on its marked ability to stand up against moisture, a powerful factor in the rusting process.

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***Testing Water Permeability of Paint Films**—With this standard apparatus a measure of the amount of water that passes through a unit of film is obtained.*

Experiments show that a straight linseed oil film allows three times as much water to pass through the film as when the same film is pigmented with Red Lead.

Water permeability tests of paint films (see illustration at lower left) show, beyond question, that Red Lead is one of the most effective metal protective pigments, because of its stubborn resistance to the passage of moisture through the film.

3. Red Lead resists solution by water—The action of water on paint films results in a partial dissolving of the film. Many metal protective films lose a considerable percentage by weight of their films through solution in water. On the other hand, the solubility losses of Red Lead paint films are practically negligible.

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feverish attempts of the industry to enlarge this fleet have met with no success due to the shortage of steel and due to a program of constructing tank cars for ammonia to assist in a fertilizer-manufacturing program sponsored by the government. An allocation of steel for a tank-car-construction program has been approved by the government and should help alleviate this shortage in 1947. There are approximately 3400 tank cars transporting liquefied petroleum gas today and there are about 2300 new tank cars on order. The majority of these new cars have been on order for some time and it is impossible now to say when their construction will be completed. Considerable effort has been devoted to stretching the movement of LP-gas in truck transports to assist in overcoming the tank-car shortage.

To overcome the inherent burden on the industry's facilities to cope with the winter requirements an increasing demand prevails for the installation of larger-volume storage capacity at all points in the distribution channels.

New Developments

The use of LP-gas for the cultivation and preservation of agricultural products has shown a healthy increase. Flame weeding passed from the development stage to that of successful commercial application. LP-gas is finding use in the many new dehydration plants. The experimental use of flame to promote the culture and ripening of products is continuing and the results appear promising.

The growing demand for the use of LP-gas in foreign countries was keenly evident throughout the year. Some petroleum companies having international organizations are now more aggresively developing these markets.

The demand for long-distance transportation of LP-gas in large quantities has accelerated plans for barge and tankship design and construction.

ASME Honors Pioneer Instrument Manufacturer

Morris Evans Leeds, pioneer in the development of modern electrical instruments, recently received the highest honor of the American Society of Mechanical Engineers, the ASME Medal. The medal was presented at the society's annual banquet in New York.

Leeds, founder and chairman of the board of Leeds & Northrup Company, Philadelphia, is the inventor of the "Leeds mechanism," a basic element in the automatically-operated balanced-current recording and controlling instruments that have become indispensable in modern industry. For his invention of this device, introduced nearly 40 years ago, the Franklin Institute honored Leeds with the Edward Longstreth Medal of Merit in 1920.

Ohio Refinery Acquired By New Organization

Western Reserve Refining Company, recently incorporated for $100,000, has taken a lease with option to purchase the plant of Erie Refining, Inc., Warren, Ohio. This plant, which was moved from the Michigan oil territory, last year, has not been operated recently due to lack of sufficient crude oil.

Thomas W. Gray has been elected president of the new company, which has made a contract with Ashland Oil & Refining Company to insure crude supply.

THE MONTH IN THE INDUSTRY

Dr. William N. Lacey To Receive Lucas Medal

Dr. William N. Lacey has been selected by American Institute of Mining and Metallurgical Engineers to receive the Anthony F. Lucas Gold Medal for 1947. The award is given for distinguished achievement in improving the technique and practice of finding and producing petroleum. Lacey was cited for "dinstinguished achievement in directing research work in the fundamentals of hydrocarbon behavior and particularly his application of these fundamentals to oil and gas reservoirs which have led to greater efficiency in oil and gas production from American fields." Last April he received the Hanlon Award for meritorious service to the natural gasoline industry for 1946.

Lacey holds A.B. and Ch.E. degrees from Stanford University and the degrees of M.S. and Ph.D. from the University of California. He went to California Institute of Technology in 1916 as an instructor, served in World War I and returned to the school in 1919 as associate professor. He became professor in 1941 and dean of graduate studies in 1946. During World War II he was member of the Advisory Board of the Chemical Corps, U. S. Army.

The Lucas Medal will be formally presented at AIME's annual meeting in New York in March.

Harts to Head NGAA Convention Committee

H. W. Harts, vice president of Warren Petroleum Corporation, Tulsa, is to head the 1947 convention program committee, of the Natural Gasoline Association of America. The convention is to be held April 23-25 in the Baker Hotel, Dallas.

Others to serve on this committee include: W. L. Bowser, The Atlantic Refining Company, Dallas; F. B. Haverfield, Continental Oil Company, Ponca City; Max Lents, J. S. Abercrombie Company, Houston; James E. Pew, Sun Oil Company, Philadelphia; W. H. Vaughan, Tide Water Associated Oil Company, Houston; and C. R. Williams, Chicago Corporation, Corpus Christi.

McCarthy Plans to Build Chemical Plant at Winnie

Immediate construction of a chemical plant at Winnie, Chambers County, Texas Gulf Coast, by McCarthy Chemical Company has been announced.

The plant's production will be aromatic and unsaturated hydrocarbon chemicals, including benzene and styrene. A company official said this will be the first of several units planned for processing petroleum and natural gas.

Winnie is the site of McCarthy's refinery and natural gasoline plant.

McCarthy Chemical Company was incorporated last October and officers other than McCarthy, include Frank B. Champion, vice president and treasurer; G. M. McGranahan, vice president and general manager, and A. G. McNeese, Jr., secretary.

Petroleum Heat and Power Acquires Taylor Refining

Through an exchange of stock Petroleum Heat & Power Company will acquire properties of Taylor Refining Company, whose 25,000-barrel refining plant is located at Corpus Christi. All its properties are valued at $8,000,000 and G. L. Rowsey, its president, will continue to direct these activites in Texas.

Other properties included are producing leases in Duval and Victoria Counties and an interest in Coastal Refineries, Inc., which has a plant at Port Isabel, as well as oil and gas production in Starr and Hidalgo Counties.

Conventions

January

17—Western Petroleum Refiners Association, technical group meeting, Washington-Youree Hotel, Shreveport.

March

2-5—American Society of Mechanical Engineers, Mayo Hotel, Tulsa.
17-19—American Institute of Mining and Metallurgical Engineers, annual meeting, Waldorf-Astoria Hotel, New York.
24-26—Western Petroleum Refiners Association, annual membership meeting, San Antonio, Texas.
24-30—Oil Heat Institute of America, Traymore Hotel, Atlantic City, New Jersey.

April

7-10—National Association of Corrosion Engineers, Palmer House, Chicago.
23-25—Natural Gasoline Association of America, Baker Hotel, Dallas.
30-2—Natural Gas Department, American Gas Association, Stevens Hotel, Chicago.

May

6-8—Petroleum Industry Electrical Association and Petroleum Electrical Supply Association, Rice Hotel, Houston.
6-8—Southwest Gas Measurement Short Course, University of Oklahoma, Norman.

September

15-17—National Butane-Propane Association, Jefferson Hotel, St. Louis.
29-3—American Gas Association, San Francisco.

Socony-Vacuum Promotes Jackson and Sheldon

R. Rea Jackson has been appointed manager of the Paulsboro, New Jersey, refinery of Socony-Vacuum Oil Company, Inc. He succeeds Howard W. Sheldon, who has been transferred to the company's headquarters at 26 Broadway, New York, to become assistant

R. REA JACKSON

Trends of Operations and Changes in Stocks

Figures on crude stocks are from Bureau of Mines weekly reports; all others from American Petroleum Institute weekly reports, which are estimates on Bureau of Mines basis.

(All figures in thousands of barrels—add 000)

Trends in Week Ended	Crude Oil			Gasoline		Gasoil and Distillate		Residual Fuel	
	Production Daily	Runs to Stills Daily	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End	Production Weekly	Stocks Week End
1945:									
January 27	4,727	4,756	221,310	14,957	88,223	4,843	33,561	9,252	51,119
February 24	4,777	4,803	219,351	15,500	95,972	4,958	28,753	9,084	46,713
March 31	4,781	4,677	223,782	14,644	98,758	4,548	26,889	9,184	41,745
April 28	4,805	4,780	223,474	14,633	94,068	4,636	28,273	9,379	39,813
May 26	4,887	4,950	222,831	15,194	89,121	4,667	29,184	9,670	38,548
June 30	4,903	4,999	220,781	15,546	86,472	4,910	32,213	9,077	40,488
July 28	4,930	4,996	218,507	16,106	86,008	4,598	36,071	9,586	42,283
August 25	4,892	4,931	211,813	15,986	84,693	4,960	39,782	9,356	46,201
September 29	4,357	3,812	222,387	11,913	79,552	3,940	43,689	7,047	46,853
October 27	4,273	4,838	224,230	15,530	74,335	5,159	43,472	8,691	45,943
November 24	4,469	4,648	219,363	15,681	83,184	4,802	45,258	8,800	47,474
December 29	4,474	4,729	218,918	14,546	95,205	5,055	36,651	8,765	42,447
1946:									
January 26	4,626	4,553	220,544	13,622	101,737	5,720	29,498	8,411	39,722
March 2	4,726	4,779	229,430	13,871	104,462	5,888	25,148	8,634	38,441
March 30	4,425	4,684	224,994	13,896	104,715	5,337	28,240	8,738	37,746
April 27	4,672	4,685	224,443	14,228	99,631	5,568	30,466	9,204	39,404
May 25	4,759	4,857	222,214	14,322	95,769	5,463	32,973	8,908	43,368
June 29	4,957	4,854	223,883	14,500	92,333	5,325	37,762	8,828	46,447
July 27	4,926	4,896	223,756	14,535	88,626	5,817	44,316	8,217	49,517
August 24	4,836	4,866	225,672	14,639	86,251	5,649	51,405	8,126	52,061
September 28	4,778	4,829	221,903	14,538	85,854	5,450	57,903	8,158	56,914
October 26	4,730	4,758	221,184	14,863	86,423	5,710	65,499	7,728	60,872
November 30	4,795	4,707	225,119	15,145	88,371	5,228	66,062	7,672	58,647
December 28	4,713	4,968	226,111	15,604	93,126	5,931	58,941	8,181	53,427

HOWARD W. SHELDON

director of Socony-Vacuum Laboratories.

Jackson has been general superintendent of the Beaumont, Texas, refinery of Magnolia Petroleum Company, an affiliate of Socony-Vacuum, for the last 10 years. He joined Magnolia as a chemist in 1923 following graduation from the University of Texas.

Sheldon was graduated from the University of Michigan in 1917 and joined Socony-Vacuum as an engineer at the Paulsboro refinery in 1926. In 1929 he was transferred to New York and became chief chemical engineer of the research and development department. Four years later he returned to Paulsboro as chemical engineer, and in 1943 became general manager of the refinery.

Sparks Named Director Esso Chemical Division

Dr. William J. Sparks has been named director of the Chemical Division of the Esso Laboratories, Standard Oil Development Company of Linden, New Jersey. He replaces Dr. Per K. Frolich, who has resigned from the company to enter other activities after 11 years as director of chemical research for the central research and technical organization of Standard Oil Company (New Jersey).

With Robert M. Thomas, now associated with the company's Baton Rouge laboratories, Dr. Sparks developed shortly before the war a new synthetic rubber, called Butyl because it was derived mainly from isobutylene.

Matteson Heads ACS California Section

Robert Matteson, supervisor in charge of the analytical division of the Richmond Laboratories of California Research Corporation, has been elected chairman of the California Section of the American Chemical Society for 1947.

Other new officers of the section are: G. K. Rollefson, University of California, vice-chairman; George M. Cook, California Research Corporation, secretary; Fred D. Tuemmler, Shell Development Company, treasurer.

Second Texas ACS Regional Meeting Stresses Refinery Technology

Chemists and engineers of the Southwest attended the Second Texas Regional Meeting of the American Chemical Society in Dallas on December 12 and 13. The meeting was sponsored by the Dallas-Fort Worth Section, of which Maurice O. Barr of Dallas is chairman. Other sections participating included Arkansas-Louisiana-Texas, Baton Rouge, Central Texas, Louisiana, Oklahoma, Panhandle Plains, South Texas, Southeast Kansas, Southeast Texas, Texas A. & M., and Texas-Louisiana-Gulf. The meeting was held in conjunction with the Texas Academy of Science. Vladimir A. Kalichevsky, chairman of the Texas-Louisiana-Gulf Section, presided at the opening session Thursday morning. Papers presented included the following:

Investigation of a Girbotol Unit Charging Cracked Refinery Gases Containing Organic Acids, by F. G. Clegg, W. E. Norris, and C. A. Murray, The Pure Oil Company, Nederland, Texas, who report that excessive diethanolamine consumption has been experienced at The Pure Oil Company's gas purifying system (Girbotol Unit) since its installation. This condition grew progressively worse as heavier hydrocarbons were charged to the system.

Investigation of the problem indicated that organic acids, principally formic and acetic, were chemically deactivating the amine. It was also found that the recommended method for determination of the amine content by titration with sulphuric acid using methyl red indicator would not give accurate results in the presence of the organic acid salts of the amine.

All attempts at regeneration of the amine solution by adding caustic to the system failed due to formation of iron hydroxide which fouled up the equipment.

Continuous and batch water washing of the feed gases was tried in actual plant operation to remove organic acids. This operation did remove the acids but resulted in a severe corrosive condition.

It was recommended that the feed gases should be scrubbed with aqueous caustic solution before entering the gas purifying system as this would remove the organic acids, thus preventing the deactivation of the amine.

A Method for the Determination of Optimum Conditions for Titrimetric Determinations was presented in two sections) I., General, by William B. Huckabay and Clyde J. Newton, and II., The Determination of Acetone, by William A. Huckabay, Clyde J. Newton, and A. V. Metler, all of Magnolia Petroleum Company, Field Research Laboratories, Dallas.

The Commercial Application of Petroleum Naphthas, by H. N. Frost and C. A. Murray, The Pure Oil Company, Nederland, Texas, discussed the uses of these hydrocarbons which are examined from the viewpoint of the consumer who has to balance volatility, solvency, odor, etc., against his individual demands. The industries reported on are in ratio to their consumption of these solvents. Also the importance and effect of selecting the correct naphtha for the industry and application when considering volatility, solvency, odor, corrosion, and sulphur are examined. Suitable solvents, diluents, and thinners for general applications are described and some data are presented to outline the future trend of this industry. The naphthas receiving most mention are those of the aliphatic branch of the family, but it is pointed out that aromatic naphthas can also be produced in quantity from war converted stills if demand requires.

The History of Commercial Production of Butadiene by the Catalytic Dehydrogenation of Butylenes, by W. L. Hiller, Jr., Cities Service Refining Corporation, Lake Charles Louisiana, dealt primarily with process developments related to the petroleum butadiene industry. The functions of the various units comprising a typical butadiene plant are given. However, the discussion is centered on the butylene dehydrogenation process beginning with the development of the cyclic steam dilution process by Standard Oil Development Company.

Among the basic design factors considered are the chemical nature of the reactant and product and the thermodynamics of the primary reaction, n-butylene—butadiene plus hydrogen.

The application of the steam dilution process using "Catalyst 1707" to commercial operation and some of the operating difficulties encountered are presented. Also discussed are the development and commercial application of continuous operating catalyst with the steam dilution process setting forth the advantages of continuous over cyclic operation.

The discussion is closed with a consideration of the possibility of future catalyst developments.

Catalyst Analysis for Catalytic Cracking of Petroleum, by A. R. Rescorla, Cities Service Refining Corporation, Lake Charles, recounted that in the evaluation of catalytic cracking processes, it has become necessary to develop several new testing procedures as well as adopt older methods of analysis. The primary purpose of this paper is to briefly summarize the various procedures employed in analyzing catalysts for catalytic cracking of petroleum, as well as to indicate the practical significance of such tests to actual operations. The data presented has been compiled from several sources of references.

The procedures for catalyst evaluation can be divided into two general groupings, namely, indirect and direct analyses. The indirect analyses consist of analyzing such properties of the catalyst as density, particle size, and chemical analysis for such common elements as Si, Fe, and Al. The direct evaluation of catalyst includes the various activity tests in which the catalyst is subjected to actual cracking in the laboratory. These analyses are commonly referred to as D+L evaluation. A series of empirical methods, such as aromatic absorption for rating catalyst activity have been developed recently.

The data is presented in such a man-

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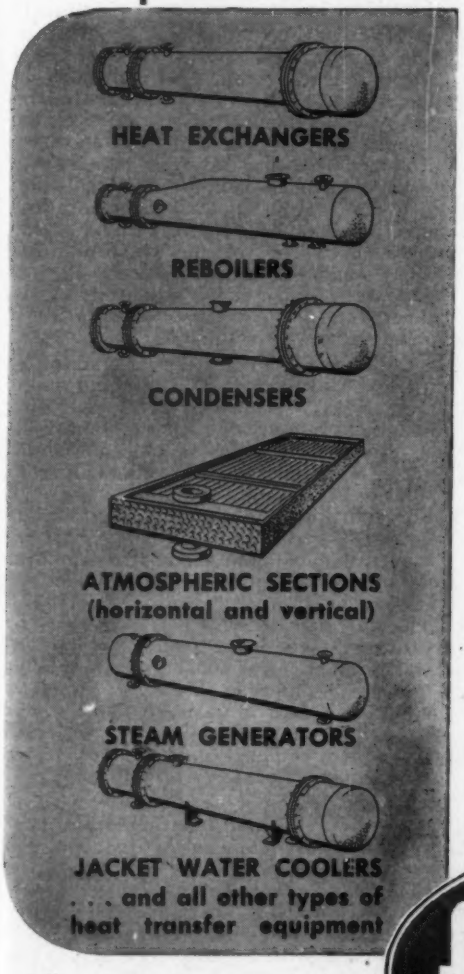

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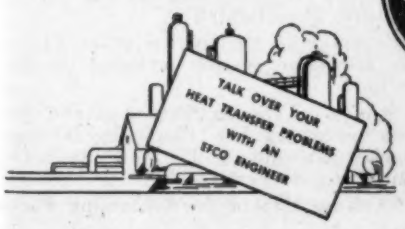

ner to indicate the relationship between the various properties of the catalyst and activity measurements. This is extended to include changes in properties of catalyst resulting from use.

In conclusion, the possible trends of catalyst analyses are outlined, and it is forecasted the number of catalyst procedures will be reduced as more data is made available; as the number of catalytic cracking units are increased, the need for catalyst evaluation tests will be emphasized, and the present laboratory cracking procedure will be improved.

Studies of Laboratory Low Temperature Fractional Distillation-Optimum Charging Rates, by C. E. Starr, Jr., J. S. Anderson, and V. M. Davidson, Standard Oil Company of New Jersey, Louisiana Division, Baton Rouge, dealt with laboratory technique, and explained that the analysis of mixtures of light hydrocarbon gases and gases such as hydrogen, nitrogen, oxygen and carbon monoxide is generally conducted first by segregation of fractions by low temperature distillation and second by analysis of the individual fractions. In such type of analytical procedure the lightest components are taken overhead from the distillation column during the charging of the sample. Published procedures for operation of low temperature distillation equipment do not ordinarily include sufficiently detailed instructions for charging samples of widely varying compositions to insure obtaining overhead fractions of maximum purity with minimum charging time.

A study has been made of charging rates for samples ordinarily encountered in petroleum refining plants. Overhead samples taken at different charging rates have been analyzed by mass spectrometer to determine the amount of contamination with higher boiling constituents. The results obtained indicate that charging rates much higher than ordinarily employed may be used with equal accuracy; use of the higher charging rates effects considerable economy of time. Supplementary data obtained also show that for special applications distillation rates may be increased several-fold over rates now generally employed.

Asphalt Emulsions

Flow Properties of Asphalt Emulsions, by D. V. Lyttleton and R. N. Traxler, The Texas Company, Port Neches, Texas, described laboratory investigations and stated that work done with the Brookfield Viscometer shows that a straight line relationship is obtained on a log-log plot of r.p.m. (rate of shear) versus absolute viscosity of asphalt emulsions. All emulsions studied fell into one of the following three groups:

1. Newtonian flow,

2. Non-Newtonian flow; reduced consistency with increasing rate of shear, and

3. Non-Newtonian flow; increased consistency with increasing rate of shear.

Each of these types are illustrated graphically. Data are given which confirm the low temperature susceptibility of asphalt emulsions.

It is pointed out that the rheological characteristics of asphalt emulsions are principally dependent on—

1. Asphalt content,

2. Particle size and size distribution, and

3, The condition of the surface of the asphalt particles as determined by the nature of the water phase.

A short discussion is given of these variables and the necessity of considering all of them. The effect of centrifuging on particle size distribution and thus on consistency is illustrated. Mention is made of the effect of stabilizers and salts on the rheological properties of bituminous emulsions.

At a luncheon talk on Thursday, T. Harrison Davies, Institute of Nuclear Studies, University of Chicago, discussed "American Proposals for Control of Atomic Energy."

Thursday afternoon, two sessions were held, with John B. Entrikin, chairman of the Arkansas-Louisiana-Texas Section, presiding over one group and with Preston L. Brandt, chairman of the Southeastern Texas Section presiding over the other group. Papers presented at the first group included:

Effect of Variables on the Alkylation of Isobutane with Ethylene in the Presence of Aluminum Bromides, by L. G. Sharp, Magnolia Petroleum Company, Dallas, in which he described the alkylation of isobutane with ethylene using eluminum bromide as catalyst to produce an alkylat containing as high as 85 percent 2.3-dimethyl-butane with complete reaction of the ethylene. The major operating variables of the reaction were studied over the following ranges:

Initial $AlBr_3$ concentration based on isobutane—0.2 to 1.0 weight percent.

Surface to volume ratio—10 to 300 square feet per cubic foot.

Ethylene partial pressure—10 to 100 pounds per square inch.

Temperature—12 to 60° C.

From an economic standpoint, the desired effect to be derived from manipulation of these variables is to produce a high quality product (high 2,3 DMB) at low catalyst consumption, and at a fast reaction rate. Fast reaction rate was favored by high values for all the variables. This was especially true for high surface where the reaction rate was increased five-fold by increasing the surface from 10 to 100 $feet^{-1}$. Further increase in surface showed no increase in reaction rate.

The alkylate was composed of from 50 to 85 percent 2,3-dimethylbutane depending upon operating conditions, the chief impurities being C_7 hydrocarbons. This C_7 material was kept low by employing high surface/volume ratio, low catalyst concentration, and low ethylene partial pressure. Reaction temperature was not significant.

Due to the relatively high cost of $AlBr_3$, prevention of catalyst consumption to form tarry complexes is especially important from an economic standpoint. Fortutiously, the conditions found to be favorable for the production of nearly pure 2,3-dimethylbutane were also the conditions found to be favorable for minimum catalyst consumption. At optimum conditions yields as high as 45 gallons of alkylate per pound of aluminum bromide consumed were obtained. Increased temperature as well as increased catalyst concentration caused increased catalyst consumption.

In addition to discussion of the reaction from a process standpoint some general considerations on the mechanism of the reaction are presented.

The Reaction of Alcohols with Dibenzolethylene, by Philip S. Bailey, and Robert E. Lutz, The University of Texas and The University of Virginia, respectively.

Relative Reactivity of Allylic Chlorides, by Lewis F. Hatch, Leon B. Gorden, and John J. Russ, The University of Texas.

Semimicro Qualitative Organic Analysis, by John B. Entrikin, Centenary College, Shreveport, made the recommendation that the tentative classification of an "unknown" be made on the basis of both its solubility in selected solvents and the elements found present since such a procedure greatly shortens the time usually required for the final identification. Representative illustrations of such classifications are given. Several tests for functional groups are discussed that have not been generally used in text books, but which have been found highly satisfactory in systematic identification work on a semi-micro scale; among these tests are the xanthate test for alcohols, the methone test for aldehydes, and the hydroxamic acid tests for esters, ethers, amides, and other classes. A systematic procedure for the separation of mixtures is summarized and its novel features are pointed out.

Removal of Acidic Compounds

The Removal of Acidic Constituents in Hydrocarbon Solutions by Absorption on Silica Gel, by D. A. Shock and Norman Hackerman, The University of Texas, described work performed in the removal and identification of the acidic compounds from the hydrocarbon phase produced by natural gas condensate wells. The usual method of adsorption in a water solution of NaOH was less effective than desired probably because these acidic constituents are present in very small quantities. Accordingly a laboratory study of the possibility of recovery of the acids by adsorption on a polar sorbent was started.

Commercial silica gel was chosen as the polar sorbent while acetic and naphthenic acids were taken as typical compounds to be removed. It was found that 300 grams of gel was capable of removing at least 10 grams of acetic acid from solutions containing 100 ppm of the acid. For 100 ppm naphthenic acid solutions in gasoline, the same amount of gel removed the acid completely from the first nine liters put through, after which the effectiveness of adsorption decreased. The gel after being eluated and reactivated gave the same result. To demonstarte the effect of concentration of solution on this method 50 gallons of 5 ppm naphthenic acid in gasoline solution was passed through the silica gel with the subsequent removal of 0.84 grams before reduced effectiveness was noticed.

The following solvents were used to elute the gel: carbon tetrachloride, benzene, amyl alcohol, isopropyl alcohol and methyl ethylketone. The last two proved the most effective. Of these methyl ethyl ketone was chosen as most feasible for practical use since it was least likely to esterify with the adsorbed acids. The possibility of inorganic eluting agents are being investigated.

Papers presented in the other Thursday afternoon session included the following:

Studies of the Activation of and Adsorption on Carbon Black, by Norman Hackerman and A. L. McClellan, The University of Texas.

"A New Method for Obtaining Parti-

THE MONTH...

cle-Size Distribution of Sub-Sieve Powders," by J. R. Musgrave and H. R. Harner, The Eagle-Picher Lead Company, a photoelectric sedimentation apparatus and necessary caluclations for determining particle size distribution are described.

By use of appropriate media and manipulation of the apparatus, the method can be used for particulate materials varying from 0.1-20 microns average radius.

The method requires only one-half to three-quarters of an hour for the complete determination and the simple calculation. It combines precision with speed and ease, and has been used with a wide variety of sub-sieve powders.

The importance is dispersion in estimating particle size is discussed.

Friday morning also the sessions were divided into two groups with Grant C. Bailey, chairman of the Oklahoma Section presiding over one group and with Norman Hackerman, chairman of the Central Texas Section conducting the other group. The following papers were presented at the first group session:

Radiography

Radiography in the Oil Industry, by J. H. Stewart, Beaumont, Texas, who explained that radiography is not new. Henri Becquerel discovered that a photographic plate in contact with pitchblende would be exposed. As a direct result of this first radiograph the Curies discovered Radium in pitchblende.

A radiograph is not a shadowgraph, nor can it be compared to one. The light and dark areas in a film vary inversely as the gamma ray coefficient. A steel casting with a porous area as a defect will show dark on the film in this area.

A carbon steel pipe lined with a stainless steel sleeve can be detected by a radiograph. The gamma ray coefficient of absorption being different for both metals will produce a light line for the carbon steel and a still lighter line for the stainless steel.

The most desirable source of gamma ray is Radium. Radium belongs to the Uranium radioactive family. Radium disintegrates emitting alpha, beta and gamma rays. Alpha particle is a Helium atom with two protons attached. The beta particle is a high velocity electron and the gamma ray is a quantum.

The gamma ray absorption coefficient varies from one substance to another. The three methods of gamma ray absorption by matter are the Compton effect, the photoelectric effect, and pair formation. (A detailed discussion is presented.)

Radium is hermetically sealed in a small silver capsule to give a constant gamma ray source. The old curie is one gram of Radium in equilibrium with Radon. The new curie is 3.7×10 disintegrations per second.

Lead absorbs gamma rays by the pair formation method and in turn gives off secondary rays. For this reason a thin sheet of lead will serve as an intensifying screen.

In taking a radiograph four variables must be considered. (1) Radium source in curies, (2) Radium to film in inches, (3) type of film used, (4) thickness of material. For instance, 200 mg. of Radium, at a Radium to film distance of 10 inches, using Eastman type "K" film with 1 inch of carbon steel would take six minutes.

The Effect of Altitude on C_4 and C_5 Blending Calculation, by N. B. Haskell and J. A. Redinger, The Texas Company, Port Arthur, Texas, described the effect of atmospheric pressure, or altitude, on calculations of the front end volatility of gasoline blends is associated with the response of gasoline fractions of varying composition to the ASTM Distillation procedure and the subsequent conversion of the distillation data from the recovered to the evaporated basis. An appartus has been devised which permits ASTM distillation tests to be conducted at any point at the atmospheric pressure of other locations. Necessary modifications to test procedure to improve reproducibility are described. A series of 16 blends containing from 0 to 30 percent C_5 component and 0 to 15 percent C_4 component was prepared and distilled at four pressures from 600 to 780 millimeters of mercury. From these data were developed the effect of atmospheric pressure and composition on distillation loss. The Sydney Young equation used for converting distillation data to other barometric pressures may be erroneous up to the 20 percent point because most of the loss occurs after reaching the initial boiling point. Existing correlations for the calculation of front end volatility of C_4 and C_5 blends are revised to incorporate the effect of barometric pressure.

Vapor Pressure Studies of Hydrogen Bromide-Aluminum Bromide-Hydrocarbon Systems, by E. R. Boedeker, R. J. Herold, and A. G. Oblad, Magnolia Petroleum Company, Dallas, stated that during the course of investigations relating to the isomerization of straight-chain paraffin hydrocarbons in the presence of aluminum bromide catalyst and hydrogen bromide promoter, it was deemed of interest to investigate the systems aluminum bromide-hydrogen bromide-hydrocarbon.

Throughout the literature pertaining to aluminum halide catalysis reference has been made repeatedly to the compound $HalX_4$ as the active complex. However, serious questions have been raised as to the existence of such compounds.

The purpose of the present work was (1) to ascertain whether the compound, $HalX_4$, was capable of existence at temperatures of 0-30° C., and (2) to obtain vapor-liquid equilibrium measurements in the above mentioned systems. These problems were investigated by a series of vapor pressure measurements.

The solubility of aluminum bromide in n-hexane was measured from 30° C. to the melting point of the solute. The solubility of aluminum bromide in n-hexane is considerably less than the ideal solubility but is slightly greater than in n-butane.

The solubility of hydrogen bromide in both n-butane and n-hexane was determined. In both cases the hydrogen bromide solubility is less than the ideal solubility and is greater in n-hexane than in n-butane.

When the solubility of hydrogen bromide in hydrocarbon solutions of aluminum bromide was determined, the solubility was found to be less than in the pure hydrocarbon.

These results show that over the range of temperatures and pressures studied, the compound, $HalBr_4$, does not exist or has so high a dissociation pressure as to be impossible to measure with the present system.

Rotation Speed in Continuous Rotary Filter Operation, by E. J. Reeves, Magnola Petroleum Company, Beaumont, developed the general equations for batch filtrations into relationship between filtrate volume and drum rotation speed in continuous rotary filter operations. Data are presented which indicate excellent agreement between calculated and observed capacity gains resulting from increased rotation speeds.

Equations are developed, also, for processing stocks which have low solid content. These relationships show that filter output is constant for a given cake thickness irrespective of the method used to maintain this thickness, i.e., recirculation of solids or reduction in rotation speed.

Mass Spectrometric Analysis

A Sample Entering System for Mass Spectrometric Analysis. Mass Spectra of Ten Aromatic Hydrocarbons, by S. E. J. Johnsen, Monsanto Chemical Company, Texas City, described a vacuum system which has been used successfully as a sample handling system in the mass spectrometric analysis of hydrocarbons up to the C_{14} members. Some use is made of sintered glass discs in contact with mercury as convenient greasless valves. The rate of pump out of heavy hydrocarbon vapors is given for three hydrocarbons. A modified form of the "Pearson" magnifying manometer is used in the system for measuring small pressures accurately. The partial cracking patterns (mass spectra) of diethyltoluene, diethylbenzene, ethyltoluene, isopropylbenzene, isopropenylbenzene, ethylbenzene, orthoxylene, styrene, toluene and benzene are also given.

In the Determination of Elemental Sulfur in Aromatic Hydrocarbons, by R. E. Lacombe, H. E. Morris, and W. H. Lane, Monsanto Chemical Company, Texas City, stated that the unreliability of the lamp method for the quantitative determination of elemental sulfur in hydrocarbons has been verified. For combined forms of sulfur, a study has been made in which certain variables of the method were investigated using a turbidimetric procedure for determination of $SO_4=$ in the absorbing solution. The method was improved by substituting Pyrex glass-wool wicks for the usual cotton ones.

A new, rapid, volumetric method for determining elemental sulfur, in the range from 0.1 to 20 percent in aromatic hydrocarbons dependent on the reaction, $S+Na_2SO_3 \rightarrow Na_2S_2O_3$, is described. Data are presented to demonstrate the accuracy of this method.

Organic Fluorine Compounds, by Ernest M. Hodnett, Oklahoma Agricultural and Mechanical College, Stillwater, related that for almost a century organic fluorides were considered to be highly unstable, and sometimes even dangerous laboratory curiosities. Recently it has been disclosed that during World War II many polyfluoro compounds were prepared and their properties investigated. These compounds are ordinarily prepared by: (1) action of silver (II) fluoride or cobalt (III) fluoride on hydrocarbons; (2) replacement of chlorine atoms by means of anhydrous hydrogen fluoride at elevated temperatures and pressures and with the aid of catalysts; and (3) replacement of chlorine atoms by means of antimony (III) fluoride. The first

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method can be used more easily now that fluorine is available in tanks. The second method is adaptable to large-scale, continuous processes. The third method can be used in any laboratory with ordinary glass equipment.

The polyfluoro compounds containing at least two fluorine atoms on one carbon possess interesting physical properties; even highly fluorinated hydrocarbons have relatively low melting points and boiling points and high stability to heat. The fluorinated hydrocarbons are chemically inert, being non-corrosive and resistant to ordinary hydrolyzing agents. These properties will make the polyfluoro organic compounds valuable as coolants, refrigerants, hydraulic fluids, boiler liquids, lubricating oils, plastic intermediates, etc. The field of fluorine chemistry is largely unexplored and offers many opportunities for profitable study.

Friday afternoon, at a joint symposium with the Texas Academy of Science, several papers of general interest were read. E. DeGolyer presented "100 Years of Geology," while W. R. Woolrich presented "100 Years of Mechanical Refrigeration," E. P. Schock "Development of Chemistry in the South," and S. W. Geyser "Fifty Years of Biology in Texas."

Friday evening a banquet was given in conjunction with The Texas Academy of Science. George Willard Watt, The University of Texas, spoke on the subject "Chemical Implications of Nuclear Energy," and Robert John Little spoke on the parallel subject, "Physical Implications of Nuclear Energy."

Chemical Society Holds Distillation Symposium

Distillation was the general theme of the thirteenth annual chemical engineering symposium of the American Chemical Society at Pittsburgh on December 30 and 31. Sponsored by the society's Division of Industrial Engineering Chemistry, of which Francis J. Curtis is chairman, the meeting was held at Mellon Institute for Industrial Research, and heard 24 technical papers.

An address on "The Gaseous Diffusion Process for Separation of Uranium Isotopes" was given at a dinner December 30, by Dr. Manson Benedict, who supervised the designing of the atomic bomb project's gaseous diffusion plant, and at present is director of process development of Hydrocarbon Research, Inc., New York.

Dr. Curtis and Dr. Edward R. Weidlein, director of the Mellon Institute and honorary chairman of the symposium, addressed the opening session, at which Dr. John R. Bowman of the Mellon Institute presided.

A general review of recent progress in distillation was given by T. J. Walsh of The Standard Oil Company (Ohio), Cleveland. Others who presented reports at the initial session were K. C. D. Hickman of Distillation Products, Inc.; Edmond S. Perry and Robert E. Fuguitt, also of Distillation Products, and Frederick E. Williams of the Hercules Powder Company.

Dr. Joseph C. Elgin of Princeton University, chairman of the symposium committee, presided Monday afternoon, when the following presented papers: F. W. Melpolder and C. W. Headington of The Atlantic Refining Company, Philadelphia; Howard S. Gardner, Ralph E. Pike, Warren E. Winsche and William J. Urbon, Massachusetts Institute of Technology and University of Rochester; Donald W. Deed and Thomas B. Drew, Columbia University; Charles B. Willingham, Vincent A. Sedlak, James W. Westhaver and Frederick D. Rossini, National Bureau of Standards, and Arthur Rose, Pennsylvania State College.

Presiding on Tuesday morning was Dr. E. W. Thiele of Standard Oil Company (Indiana). Those who gave reports at this session were: M. R. Fenske and C. S. Carlson, Standard Oil Development Company; Dorothy Quiggle, Pennsylvania State College; John Griswold, E. I. du Pont de Nemours & Company; J. W. Morris, University of Texas; T. S. Mertes, J. A. Gerster and A. P. Colburn, University of Delaware; C. K. Buell and R. G. Boatright, Phillips Petroleum Company, and Dr. Bowman and R. C. Briant, Mellon Institute.

Authors of papers heard at the final session Tuesday afternoon, at which Dr. W. L. McCabe of the Carnegie Institute of Technology presided were: J. A. Weedman and B. F. Dodge, Yale University; Walter E. Lobo and Brymer Williams, M. W. Kellogg Company; J. G. Aston, Pennesylnavia State College; Wolcott Dennis, Air Reduction Company; Dudley A. Williams; H. O. McMahon, Arthur D. Little, Inc.; W. S. Forsythe, T. G. Stack, J. E. Wolf and A. L. Conn, Standard Oil Company (Indiana); Whiting; John Griswold, E. I. du Pont de Nemours & Company; and P. B. Stewart, University of Texas.

Also presented, by title, were papers by Arthur Rose, Pennsylvania State College; James A. Davies, The Texas Company, and Dr. Bowman.

Automotive Engineers Discuss Fuels and Rubber

The annual meeting of the Society of Automotive Engineers at Detroit January 6-10 had before it papers covering a variety of material important to engineers and designers concerned with aircraft, diesel engines, fuels and lubricants, passenger cars, tractors, trucks, and busses. Papers especially interesting to petroleum refining industry include:

A Discussion of Antiknock Performance Related to ASTM and Research Octane Numbers and the Development of Optimum Fuel-Engine Relationships, presented by Errol J. Gay and Henry T. Mueller, of Technical Service Department, Ethyl Corporation.

Methods of Using Mean Specific Heats to Calculate the Thermo-Dynamic characteristics of the working media of gas-turbine powerplants, presented by Newman A. Hall of United Aircraft Corporation, East Hartford, Connecticut. Dr. Hall also presented newly developed charts showing mean specific heats of air, with correction factors for water vapor, fuel vapor, and products of combustion.

"Americans should make their own rubber," asserted J. E. Hale of The Firestone Tire and Rubber Company. He said that further technical development will produce tires, tubes, and other rubber goods equal or superior to those made from natural rubber, and insisted that wartime synthetic-rubber plants should be placed in operation under American commercial control to avoid price fluctuations and to insure independence from foreign natural rubber.

Mutual Adaptation of Fuels and Engines was the subject of a discussion by S. D. Heron, consulting engineer, New York, who stated that satisfactory performance is dependent on technical cooperation between the aircraft power plant, steel, and petroleum industries.

J. G. Moxey, Jr., of Sun Oil Company, Marcus Hook, Pennsylvania, warned that as a result of the wider use of catalytic cracking in the refining of moter fuels, the trend is toward lower volatilities, and engine manufacturers should expect and prepare for some difficulties with starting in cold weather.

Election of C. E. Frudden, of the Tractor Division, Allis-Chalmers Manufacturing Company, Milwaukee, as the 1947 president of the Society of Automotive Engineers was announced at the business session held January 7. Frudden succeeds L. Ray Buckendale, of Timkin-Detroit Axle Company.

Thomas President-Elect of American Chemical Society

Dr. Charles A. Thomas, vice president and technical director of Monsanto Chemical Company, St. Louis, and project director of the Monsanto-operated Clinton Laboratories at Oak Ridge, Tennessee, has been elected president of the American Chemical Society for 1948.

Dr. Thomas became president-elect of the society on January 1, 1947, when Professor W. Albert Noyes, Jr., chairman of the chemistry department of the University of Rochester, became president, succeeding Bradley Dewey. Dr. Thomas was chosen by the society's council from four nominees receiving the largest number of votes in a national mail ballot of the society's 48,000 members.

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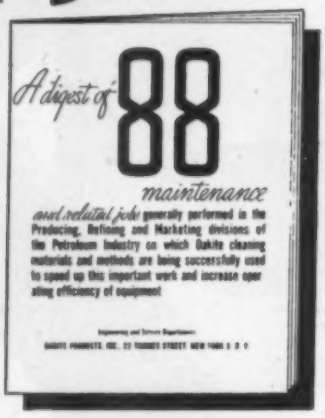

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Commission, Dr. Thomas is one of the five co-authors of the Acheson-Lilienthal report on international control of nuclear power which was prepared for the Secretary of State's Committee on Atomic Energy early this year. During World War II, he was an active participant in the development of the automic bomb, and in the production of catapult rockets to get American models of German robot bombs into the air.

In addition to his close association with nuclear research, Dr. Thomas has been active in peacetime investigations of far-reaching industrial and scientific importance, including the development of Ethyl gasoline and the extraction of bromine from sea water.

Dr. Thomas was graduated from Transylvania College, Lexington, Kentucky, with the degree of bachelor of arts in 1920. He received the master of science degree from Massachusetts Institute of Technology in 1924, and the doctor of science degree from Transylvania in 1933.

In 1923 he joined General Motors Research Corporation as a research chemist at Dayton, Ohio, participating in studies which resulted in the discovery of tetraethyl lead as an antiknock agent in motor fuels, and in the recovery for the first time in history of minerals from the ocean.

Later, he became a research chemist on the staff of the Ethyl Gasoline Corporation. In 1926, with Dr. Carroll A. Hochwalt, he established the Thomas and Hochwalt Laboratories at Dayton, specializing in consulting research work for industrial organizations. In 1936, the laboratories were acquired by the Monsanto Chemical Company and Dr. Thomas became central research director of the company, with Dr. Hochwalt as associate director.

During this period Dr. Thomas also served for a time as vice president in charge of research of Sharples Corporation. His work for Monsanto was concerned chiefly with synthetic resins from petroleum hydrocarbons, the formation of diolefins, olefins and aromatics by the pyrolysis of hydrocarbons, plastics, and the synthesis of various aliphatic compounds.

In April, 1945, Dr. Thomas became vice president of Monsanto, and in September of that year he was named technical director of the company and a member of its four-man executive committee.

During World War II, Dr. Thomas had charge of the final purification and metallurgy of plutonium, element 94, for use in atomic bombs. The Clinton Laboratories at Oak Ridge, where chemists and physicists produced the first experimental quantities of plutonium, were placed under his general direction on July 1, 1945. After the war, Dr. Thomas continued in charge of the laboratories as work was begun on the conversion of atomic energy to peacetime uses.

Dr. Thomas was awarded the Medal of Merit by Secretary of War Robert P. Patterson last March for his work on the bomb project.

Author of numerous scientific papers and holder of several patents in the United States and abroad, most of them in the field of organic chemistry, Dr. Thomas wrote a monograph on "Anhydrous Aluminum Chloride in Organic Chemistry" which became a standard work in that field.

He has been a director-at-large of the American Chemical Society since 1942 and was chairman of its Dayton Section from 1930 to 1932. He is a member of the board of editors of the American Chemical Society Monographs. He is a fellow of the American Association for the Advancement of Science, the American Institute of Chemists, and the Chemical Society of London; a director of the board of governors of the National Farm Chemurgic Council, and a member of the American Institute of Chemical Engineers and Alpha Chi Sigma.

CNGA Will Discuss Process Control at February Meeting

Automatic control of light-hydrocarbon processing equipment producing closely-fractionated products will be discussed at the February meeting of California Natural Gasoline Association. Processes to be discussed by E. H. Reynolds, William Troutman and Gordon Lawn of Richfield Oil Corporation, will vary from single-column processes operated for production of a constant-vapor-pressure bottoms product to multiple-column processes operated for production of pure hydrocarbons and narrow-boiling-range cuts. The authors will present their opinions upon such important problems as selection of the controlled variables, selection of locations for measuring the controlled variables, and determination of throttling range and automatic reset settings.

The meeting will be held at Rio Hondo Golf Club, on Thursday, February 6, at 6 p.m.

API Public-Relations Program Goes Into Action

Activity of the public-relations program for the petroleum industry got underway in December, when the public-relations committee of the American Petroleum Institute held its first meeting as an enlarged group of which John M. Lovejoy, Seaboard Oil Company, is chairman. This committee reviewed and approved the operating plan of the advisory sub-committee on public relations, which was designated as the operating committee with Robert T. Haslam, Standard Oil Company (New Jersey), chairman; Franklyn Waltman, Sun Oil Company, and Ralph Champlin, Ethyl Corporation, vice chairmen.

Other members of this committee are: J. L. Dupree, 405 Lexington Avenue, New York; H. L. Curtis, Shell Oil Company, New York; George H. Freyermuth, Standard Oil Company (New Jersey), New York; Don E. Gilman, Western Oil & Gas Association, Los Angeles; W. R. Huber, Gulf Oil Corporation, Pittsburgh; C. R. Musgrave, Phillips Petroleum Company, Bartlesville; Conger Reynolds, Standard Oil Company (Indiana), Chicago; Richard Rollins, The Atlantic Refining Company, Philadelphia; Haines Finnell, Union Oil Company, Los Angeles; Merle Thorpe, Cities Service Oil Company, New York; Edgar A. Waite, Standard Oil Company of California, San Francisco; Douglas Campbell, The Pure Oil Company, Chicago; John Wieland, The Standard Oil Company (Ohio), Cleveland.

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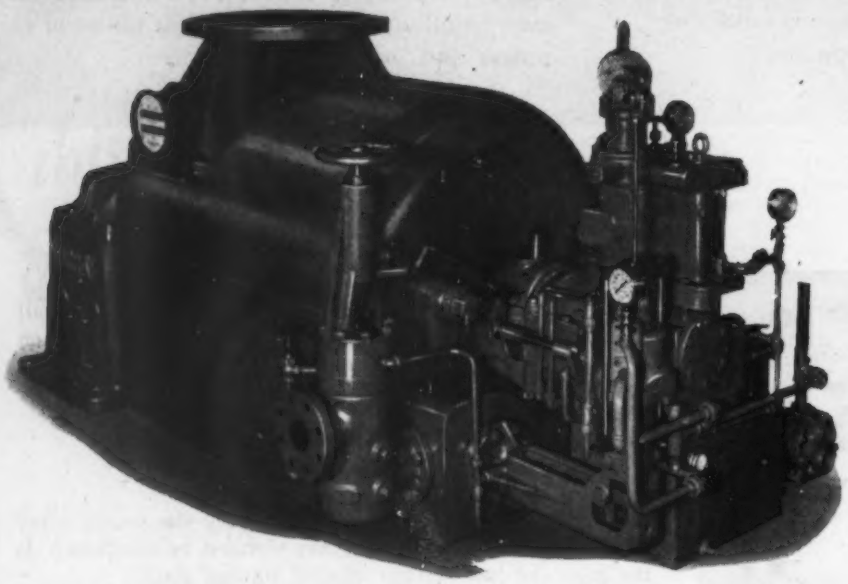

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relations counsel, will manage the campaign under direction of the operating committee.

Organization in the field got under way through the selection of five temporary organizing committees, which are to serve in perfecting permanent organizations. Chairmen of these organizing committees are: Robert H. Colley, The Atlantic Refining Company, Philadelphia; L. S. Wescoat, The Pure Oil Company, Chicago; Jake L. Hamon, Cox & Hamon, Dallas; W. H. Ferguson, Continental Oil Company, Denver; Reese H. Taylor, Union Oil Company, Los Angeles.

In his letter to these chairmen Lovejoy said that in each district the permanent organization should be large enough so that every phase of the industry and related groups, such as natural gas, natural gasoline and suppliers, may be represented. As to workings of the program, he added:

"The committee set-up will be a two-way street. The public relations committee, which will direct the program under the chairmanship of R. T. Haslam, will supply material, facts, and ideas to the district committees and in turn obtain ideas and inspiration from the district committees."

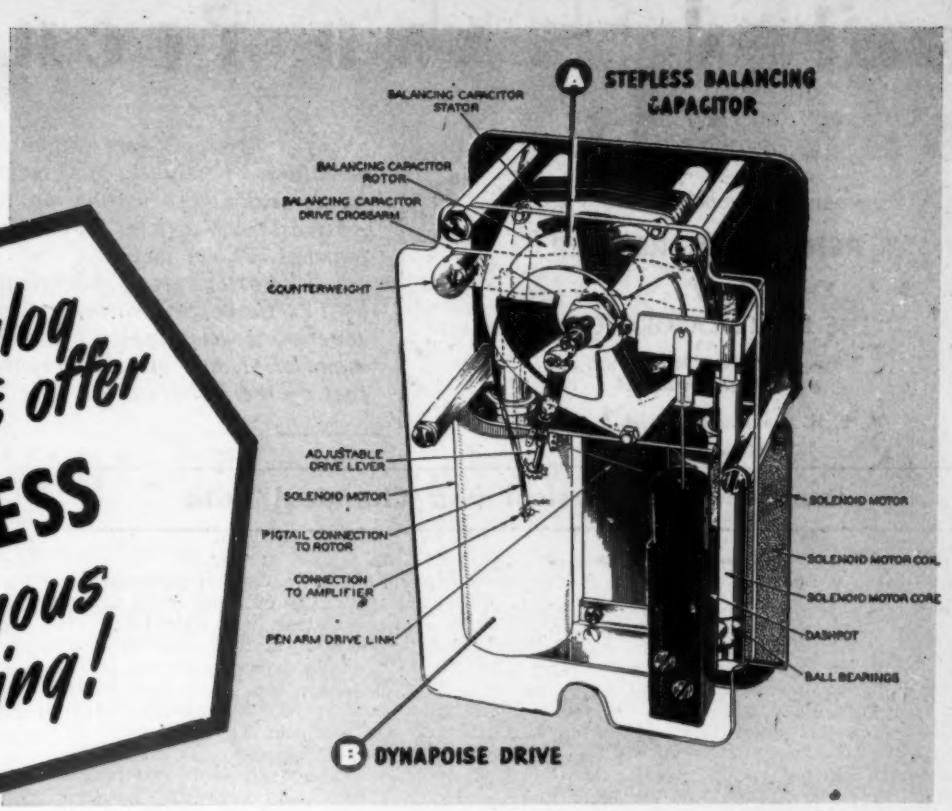
He cited results of the nation-wide poll as evidence of the need for a public-relations program, saying:

"We feel that the first step in our program is to show the oil industry itself the need for a public-relations program. We are advised that the results of the public-opinion survey recently concluded will be available for distribution with the next month or six weeks. As you know, the survey indicates that more than one third of the people of the United States believe the oil industry to be monopolistic in nature and that 57 percent are willing to believe that prices for our products are not representative of the free competitive market which actually exists. It seems to us that this alone is sufficient reason for the industry to make a serious attempt to correct this misconception. The second step in the program is to reach the public with the proper story of our stewardship of this great industry. This can not be done effectively without the support of the thousands of companies and individuals directly engaged in the industry."

Restrictions Removed On Tetraethyl Lead

Although restrictions on the production and use of tetraethyl lead were removed last month, short supply brought this analysis of the situation from Earle W. Webb, president of Ethyl Corporation:

"During the period of governmental controls, inventories of metallic lead and tetraethyl lead in our manufacturing plants have been necessarily reduced to rock bottom. Metallic lead is still short. It will be necessary to obtain increased supplies of it before we can assure our customers of all the antiknock compound they will want. We shall do our utmost to secure enough lead to meet these requirements as quickly as possible. In the meantime, any sudden increase in orders will be unfortunate."



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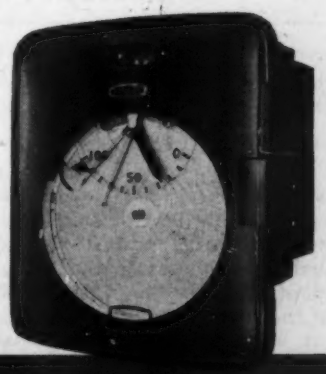

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The abstracts here presented are selected from the current literature of science and technology to afford reference to fundamental information not easily available to all readers. Abstracts of articles appearing in readily obtainable trade journals are not included.

Photostat copies of original articles will be supplied at cost by The Leslie Laboratories. Complete or limited bibliographies covering special topics by title, by abstracts, or in complete manuscript, will also be prepared and furnished at reasonable cost by the Laboratories.

Fundamental Physical and Chemical Data

The Thermodynamics of Styrene and Its Methyl Derivatives. CHARLES W. BECKETT AND KENNETH S. PITZER. *Jour. Amer. Chem. Soc.*, **68** (1946) pp. 2213-14.

Details were given in a preceding paper that are necessary to calculate the thermodynamic properties of styrene in the perfect-gas state. The authors calculate the thermodynamic properties of styrenes and present the results in tabular form. The heat of dehydrogenation of ethylbenzene to styrene is also calculated. The thermodynamic functions of some of the methyl styrenes were estimated by the method of increments, and the results of these calculations are also given in tabular form.

The Heat Capacity, Heats of Fusion and Vaporization, Vapor Pressure, Entropy, Vibration Frequencies and Barrier to Internal Rotation of Styrene. KENNETH S. PITZER, LESTER GUTTMAN AND EDGAR G. WESTRUM, JR. *Jour. Amer. Chem. Soc.*, **68**, (1946) pp. 2209-12.

Styrene is one of the most important polymerizing monomers and its fundamental thermodynamic properties are of considerable importance. The authors have published a preliminary summary on this subject. The directly measured data are presented in detail in the present paper. The heat capacity of solid and liquid styrene was measured from 14 to 300° K. and the heat of fusion found to be 2617 cal./mole at 242.47° K. (the melting point). The vapor pressure was measured from 0 to 60° and from it the heat of vaporization was calculated as 10,500 cal./mole at 25° C. The entropy at 298.16° K. was calculated as 56.78 cal./deg. mole for the liquid and 82.48 cal./deg. mole for the hypothetical ideal gas at 1 atmosphere. An assignment of the vibration frequencies was made and the entropy calculated from statistical mechanics. It was necessary to assume a 2200 cal./mole potential barrier to obtain agreement with the experimental value. The meaning of this barrier in terms of conjugation or resonance energy is discussed.

The Dipole Moment of 1-Nitrobutane. JOHN G. MILLER AND HENRY S. ANGEL. *Jour. Amer. Chem. Soc.*, **68** (1946) p. 2358.

The values of the dipole moments of the nitroparaffins in solution are important reference standards for the estimation of the effect of resonance in nitrocompounds that cannot be studied in the gaseous state. Such data as are available are reviewed. Wide variations in the reported values led the authors to measure the dipole moment of 1-nitrobutane, using benzene as solvent in order to conform with previous work. The value of the moment at 25° C. is 3.40 ± 0.01 D. The authors believe from a study of all available data that 3.4 D is the correct figure for the solution value of the nitrogroup moment in these compounds.

Manufacture: Processes and Plant

Gasoline Loss Control and Molecular Weight Determinations. H. G. KOEFOED. *Jour. The Institute of Petroleum*, **32** (1946) 529-74.

All handling of gasoline during refining, storage, transport, or distribution causes losses that may be small or large, depending upon the method of handling, the quality of the gasoline, and the efficiency of control. The author believes that if the loss does not exceed 1.5 percent it can be considered normal for proper equipment that is carefully used. Loss through waste and negligence are considered to be the most conspicuous. Losses resulting from changes in temperature are considered. Loss through evaporation is by far the largest and most difficult to overcome. Analyses of hydrocarbon vapors expelled from tanks is given and methods of analysis for quickly ascertaining the gasoline-vapor content in an air-gasoline mixture are considered. The apparatus and procedure is described in some detail. Examples are given showing the application of the methods of analysis presented. The filling of tank cars is taken as a good example. Filling and breathing losses are considered in some detail. Phenomena occurring in the use of a typical tank are discussed and data are given. The measurements of tank temperatures is considered and procedures for combatting breathing losses are discussed.

The Production of Lubricating Oils from the Olefines Produced in the Fischer-Tropsch Synthesis. D. GALL. *Jour. Soc. Chem. Ind.*, **65** (1946) pp. 185-9.

Since 1933 considerable attention has been paid in Germany to the possibility of producing lubricating oils by the polymerization of olefines found in (or prepared from) the primary products of the hydrocarbon synthesis process of Fischer-Tropsch. The author studied the preparation of lubricating oils by the $AlCl_3$ polymerization of the olefines present in the primary product obtained by atmospheric pressure synthesis from carban monoxide and hydrogen in the ratio 1:2 over a cobalt catalyst. The effect of varying temperature, catalyst and boiling range of the starting material on the reaction and its products is described. The oils obtained possess properties that approximate to the requirements of the Air Ministry Specification D.T.D. 109 for aircraft lubricants in all respects except the increase in viscosity on oxidation. It was found possible to overcome this objection by subjecting the oils to a carefully controlled hydrogenation in the presence of molybdenum disulphide. The data secured in the course of the work are presented in some detail in tabular and graphical form and a bibliography of 17 references is included.

Azeotropic Purification of Styrene. LLOYD BERG, J. M. HARRISON, AND C. W. MONTGOMERY. *Ind. & Eng. Chem.* **38** (1946) pp. 1149-52.

Styrene usually is manufactured by the dehydrogenation of ethylbenzene. Yields per pass of styrene are reported from 30 to 80 percent. Partial conversion does not represent a loss for styrene can be readily separated from ethylbenzene and the latter recovered for recycling. The separation of styrene from ethyl-

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Di-o-tolyl zinc
3-Chlorotetrahydrofuran
2,2′,2″-Tripyridyl
2,2-Dimethyl-1,3-propylene glycol
2-Hydroxy isovaleric acid
Behenolic acid
Stearolic acid
3-Amino-2,4-dimethylbenzoic acid
Cyclobutadiene
2-Methyl-1,3-propylene glycol
1,3-Pentanediol
2-Methyl-1,2-butylene glycol
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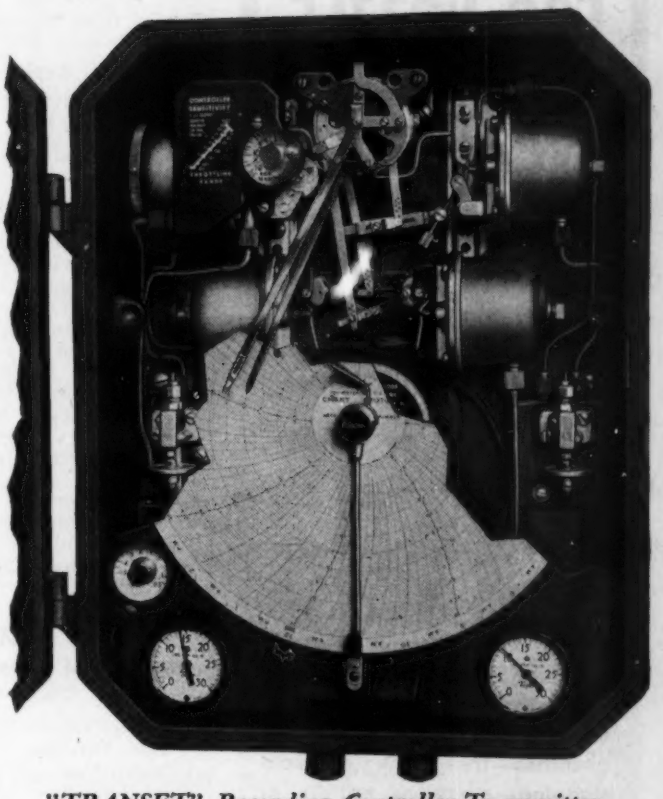

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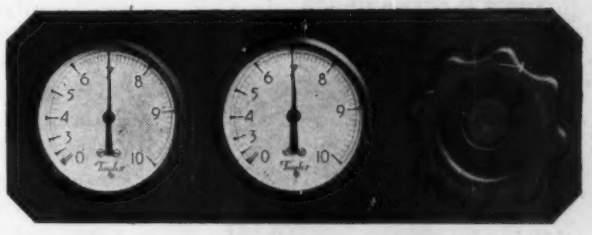

Indicating Receiver (left). Set Pointer (center). Set Point Adjustment (right).

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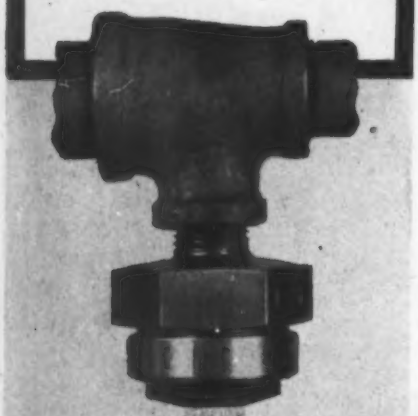

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benzene can be accomplished by the use of entrainers such as 1-nitropropane, the lower aliphatic monobasic acids, a number of alcohols of more than two carbon atoms, methyl-Cellosolve, or water. Entrainers can be recovered by phase separation, solvent extraction, and pressure rectification. The best entrainers appear to be intermediate boiling compounds, such as isobutanol or 1-nitropropane. Azeotropic methods enhance the separation of styrene from ethylbenzene with respect to the quantity of styrene recovered in a given column, or with respect to the saving of column capacity and heat for any given purity. By means of a combination of ordinary rectification and azeotropic distillation, any hydrocarbon impurity can be removed from styrene.

Practical Aspects in Design of Liquid Agitators. RICHARD E. CHADBOCK. *Chemical Engineering* **53** (1946) pp. 51-4.

Notwithstanding all that has been written on the subject of the design of agitators for liquids, and mixtures of liquids with solids or gases, the subject must be regarded still as far from exact. Practical experience is necessary in many instances to insure a correct design without resort to theories or formulas of doubtful validity. The author presents such a system of selection, that has had the benefit of much actual and successful use. A diagram is given showing four types of agitators from which a selection can be made in 95 percent of the liquid-mixing problems. Small modifications of these types handle most other problems. Agitator impeller types are discussed, as well as the power consumption and design details of agitators. A bibliography of 6 references is included.

Directory of Materials for the Construction of Chemical Equipment. *Chemical Engineering* **53** (1946) pp. 110-19.

The Directory of Materials for the Construction of Chemical Equipment comprises part of Chemical Engineering's Twelfth Report on Materials of Construction and affords a quick means for identifying materials available for the construction of process equipment. Approximately 600 items are included in the listing. Materials are arranged alphabetically within each of seven classes of material, including metals and alloys, carbon and graphite, cement, mortar and putty, ceramic materials, plastics, rubber and wood. The name of the manufacturer of the material is given as well as the nominal chemical composition. Forms in which the material is available are given and the services for which it is intended are indicated.

Chemical Resistance of Constructional Metals and Non-Metals. *Chemical Engineering* **53** (1946) pp. 120-50.

The editors of Chemical Engineering have compiled a table showing the resistance of materials to 24 common and troublesome chemicals. Some of the important applications are indicated for which the materials are used. However, it is noted that corrosion involves many more variables than this or any table can possibly take into account. Thus, the data presented are not to be considered more than an aid in narrowing the field of materials that are worth investigation in any particular instance.

Chemical and Heat Resistance of Gasket Materials. H. H. DUNKLE AND E. C. FETTER, *Chemical Engineering*, **53** (1946) pp. 102-9.

Selection of gaskets offers special problems in material choice as well as in type of construction. Resistance to heat and corrosion are the two most important of many factors that enter into the choosing of a gasket material. The article considers gasket constructions that are commonly used in the process industry. It is sometimes assumed that a material suitably resistant when used as the construction material for a heavy reaction vessel will also be satisfactory as a gasket under the same conditions. Too frequently this is not the case. Metallic and non-metallic gasket materials are considered in some detail. A table summarizing the chemical resistance of gasket materials is presented. Resistance of various metallic gaskets and gaskets made of asbestos, rubber and miscellaneous materials is given for a long list of substance against which protection is required.

Chemical Composition and Reactions

Primary Process in Pyrolysis of Pentenes. EVERETT GORIN, A. G. OBLAD, AND R. F. SCHMUCK. *Ind. & Eng. Chem.* **38** (1946) pp. 1187-93.

The authors investigated the pyrolysis of pentenes and pentane-pentene mixtures for the production of diolefins in an effort to determine the optimum conditions for the production of dienes. Butadiene is the principal diolefin obtained from the pyrolysis of 1-pentene and 2-pentene. Small amounts of 1,3-pentadiene are produced at the same time in the pyrolysis of 2-pentene. The highest yields of butadiene are obtained from 2-pentene at 750-800° C. and at pressures below 0.2 atmosphere. The main side-reaction in the pyrolysis of the normal pentenes is cracking to low-molecular-weight olefins. Isoprene is the principal diolefin from the pyrolysis of tertiary pentenes. A small quantity of butadiene is produced at the same time. The best yields of isoprene are obtained by pyrolysis of trimethylene at 800° C. The paraffinic components in pentane-pentene mixtures do not contribute appreciably to the production of diolefins in the cracking of these mixtures. The primary reactions in the decomposition of the pentanes were determined by extrapolation of the experimental results. Comparison was made with the reactions predicted by Rice by the free radical chain hypothesis. The theory of Rice must be modified to account for all of the primary reactions observed.

Ethylbenzene. ALFRED W. FRANCIS AND E. EMMET REID. *Ind. & Eng. Chem.* **38** (1946) pp. 1194-1203.

Ethylbenzene is produced on a large scale by the liquid-phase alkylation of benzene with ethylene in the presence of aluminum chloride. The authors report that the ethylation of benzene with ethylene has been controlled to give yields of monoethylbenzene about 15

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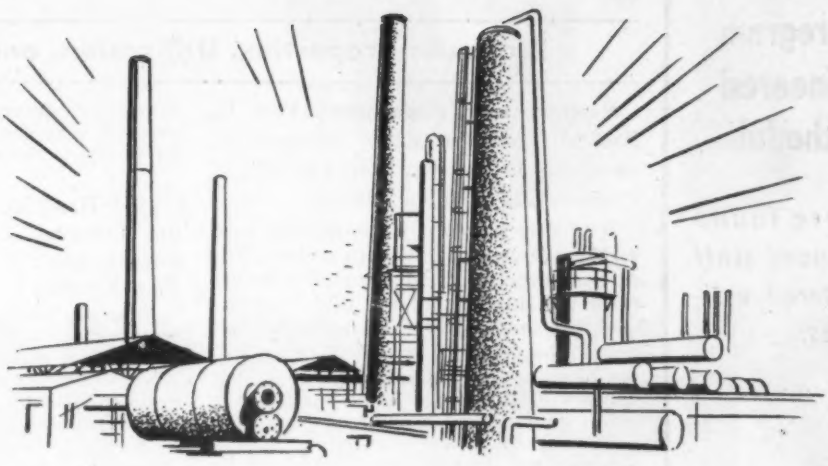

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percent higher than those reported by most other investigators at corresponding mole ratios of the reacting substances. Reaction conditions are 100° C. and a pressure of 100 to 300 pounds of ethylene applied above the catalyst. Under these conditions the reaction rate is increased nearly a hundred fold over some industrial processes, so that a continuous operation is practical. The authors correlated the yields of ethylbenzene as reported in 60 investigations of others, and show the relationship of mole ratios of reagents to reaction conditions. These are compared to theoretical curves for multiple-stage reactions with a common reagent. The ethylation of benzene takes place at the same rate as the ethylation of ethylbenzene and other partly ethylized benzenes, although this is contrary to common opinion. Some observations can be best explained by assuming that under suitable conditions the ethylation reaction is practically instantaneous. The reaction conditions are given for making pure ethylbenzene from ethylene mixed with paraffin gases, or from a low concentration of benzene in a mixture with non-aromatic hydrocarbons. The data are presented in some detail in tabular and graphical form and a bibliography of 104 references is included.

Products: Properties, Utilization, and Analysis

Researches on Asphaltenes. Part II. Radical Reactions. F. J. NELLENSTEYN, PH.D., AND J. DORLEYN, *Jour. The Inst. of Petroleum*, **32** (1946) pp. 582-86.

Asphaltic bitumen is considered as a highly protected oleosole of carbon. The carbon kernels with the protective bodies form the micels, and the oily constituents, the medium of these colloidal systems. The colloidal structure of asphaltic bitumen is generally accepted but views still differ widely as regards the inner structure of the micel. Some believe that the micel consists of highly polymerized hydrocarbon, combined by sulphur and oxygen bridges. The author, however, believes that this constitution is improbable on account of the fact that a polymer characteristic of asphaltic bitumen has never been proved and the fact that the oxidation of asphaltic bitumen micel with potassium permanganate yields mellitic acid. Results of experimental work on the chlorination of asphaltic bitumen are described and discussed. The authors believe that the formation of C_6Cl_6 is definite proof of the presence of mono atomic radicals in asphaltic bitumen.

Evaluation of Laboratory Distillation Apparatus. FRANK C. COLLINS AND VERNON LANTZ. *Ind. & Eng. Chem., Anal. Ed.*, **18** (1946) pp. 673-77.

The authors note that the usual practice of evaluating distillation apparatus under idealized conditions of total reflux may lead to questionable results in actual operation at finite reflux ratios. Actual separations obtained at finite reflux ratios are generaly appreciably lower than would be predicted from column efficiency measured at total reflux. Test methods were developed for evaluating laboratory distillation equipment under conditions approximating actual operation rather than ideal conditions for maximum separation. Test methods for evaluating the other principal characteristics of distillation apparatus are also described in detail. The authors found that the Oldershaw bubble-plate column gave exceptionally high throughput capacity and a low operating holdup per theoretical plate as compared to other laboratory columns. The Oldershaw glass bubble-plate column was improved and studied, using the test methods developed. The column was found to be highly stasifactory for analytical distillations of hydrocarbon solutions because of uniformity of its operating characteristics. Automatic still heads and other accessory equipment also have been developed that contribute to the uniformity of the performance of the column.

New Head for Laboratory Fractionating Columns. J. D. BARTLESON, A. L. CONRAD, AND P. S. FAY. *Ind. & Eng. Anal. Ed.*, **18** (1946) p. 724.

Several take-off heads capable of maintaining a constant reflux ratio regardless of variations in distillation rate have been described in the literature, but none of these devices is satisfactory for the routine laboratory distillation of petroleum products in which appreciable amounts of butane hydrocarbons are to be measured. The low-boiling components vaporize in the take-off line and re-enter the column. The device described by the authors overcomes this difficulty and has the further advantage of high throughput and low holdup. A detailed description and drawings of the head are presented.

Some Observations on the Development of Extreme Pressure (E.P.) Properties by Chlorine and Sulphur Additives in Mineral Oil, as Assessed by the Four-Ball Machine. W. DAVEY, *Jour. Inst. of Petroleum*, **32** (1946) pp. 575-81.

The author studied the E.P. properties shown when chlorine and sulphur additives are used. In general the results confirm previously published work. The four-ball machine was used in the investigation. The use of these additives is judged as being justified under high-load conditions. The formation of ferrous chloride films by chlorine additives was confirmed and the effect of sulphide films and hydrogen chloride studied. The breakdown of the additives to form hydrogen chloride is believed to be just sufficient to produce a film of the requisite thickness to carry the applied load. The chloride film is probably produced by the formation of an addition complex by the chlorine additive and the iron surface, followed by the breakdown of this complex to form a ferrous chloride film, and then by the formation of hydrogen chloride.

Proportional Flow Controller for Liquids. WILLIAM R. WILKINSON AND HAROLD A. BEATTY, *Ind. & Eng. Chem., Anal. Ed.*, **18** (1946) pp. 725-6.

An old problem in fractional distillation is the accurate control of reflux ratio with variable throughput. A proportional flow controller was developed and used successfully in a 5-inch dia-

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meter column for one year. It operates on the principle of directing a downward flow of liquid into a uniform, free-falling sheet or curtain that is split into two parts by a movable divider. The apparatus is described and sketches are presented. The device was tested at reflux ratios in the range from 1 to 30. The ratios predicted from the position of the divider were obtained with an accuracy varying from ±1 per cent at a ratio of 1 to ±10 at a ratio of 30, throughout the entire range of 5 to 25 gallons per hour flow. The device has no moving parts, is continuous rather than intermittent in operation, and has a relatively low holdup.

Determination of Extinction Corrections in Infrared Analysis of Gaseous Hydrocarbon Mixtures. J. H. Lee, *Ind. & Eng. Chem., Anal. Ed.*, 18 (1946) pp. 659-61.

Infrared absorption spectrophotometry is acquiring an important position in the routine testing of light hydrocarbon mixture. Speed is an important requirement in the methods, and so loss of time in computing the results of infrared analysis, short procedures for such calculations become of considerable imporance. Infrared analysis of gaseous hydrocarbon mixtures involves determination of the extinctions for the individual components. Data must be corrected for second-order effects caused by energy scattered within the instrument, the structure of the absorption band, and intermolecular action. A simple relationship permits correction for these deviations. This method simplifies the work connected with the calibration or recalibration of the spectrophotometer. Use of a reference substance as a diluting agent is suggested in cases in which molecular interaction influences absorption.

Determination of Olefinic Unsaturation. G. R. Bond, Jr., *Ind. & Eng. Chem., Anal. Ed.*, 18 (1946) pp. 692-96.

The determination of olefinic unsaturation in hydrocarbon solutions has long been a controversial subject in the petroleum industry. A method that will give a direct volumetric measurement of the olefiinic unsaturation regardless of the type of olefins present, and which is without effect on paraffins, naphthenes, or aromatics is the ideal solution of the problem. The authors state that such a method has been developed and has given most promising results on a wide variety of samples, both synthetic and natural. It can be used for determining olefins in dark-colored samples. The procedure is based on the reaction of nitrogen tetroxide with olefinic materials in order to convert them into oily addition products, or nitrosates. Separation of the material that does not react, including paraffins, naphthenes, and aromatics, can be effected by steam-distillation or by reaction and solution of the nitrosates in alcoholic potassium hydroxide and potassium sulfide. The change in volume of the sample gives the measurement of the olefinic unsaturation.

Picric Acid Method for Determination of Aromatic Content of Aviation Gasoline. C. M. Gambrill and J. B. Martin, *Ind. & Eng. Chem., Anal. Ed.*, 18 (1946) pp. 689-92.

The difference in solubility of picric acid in aromatic and non-aromatic hy-

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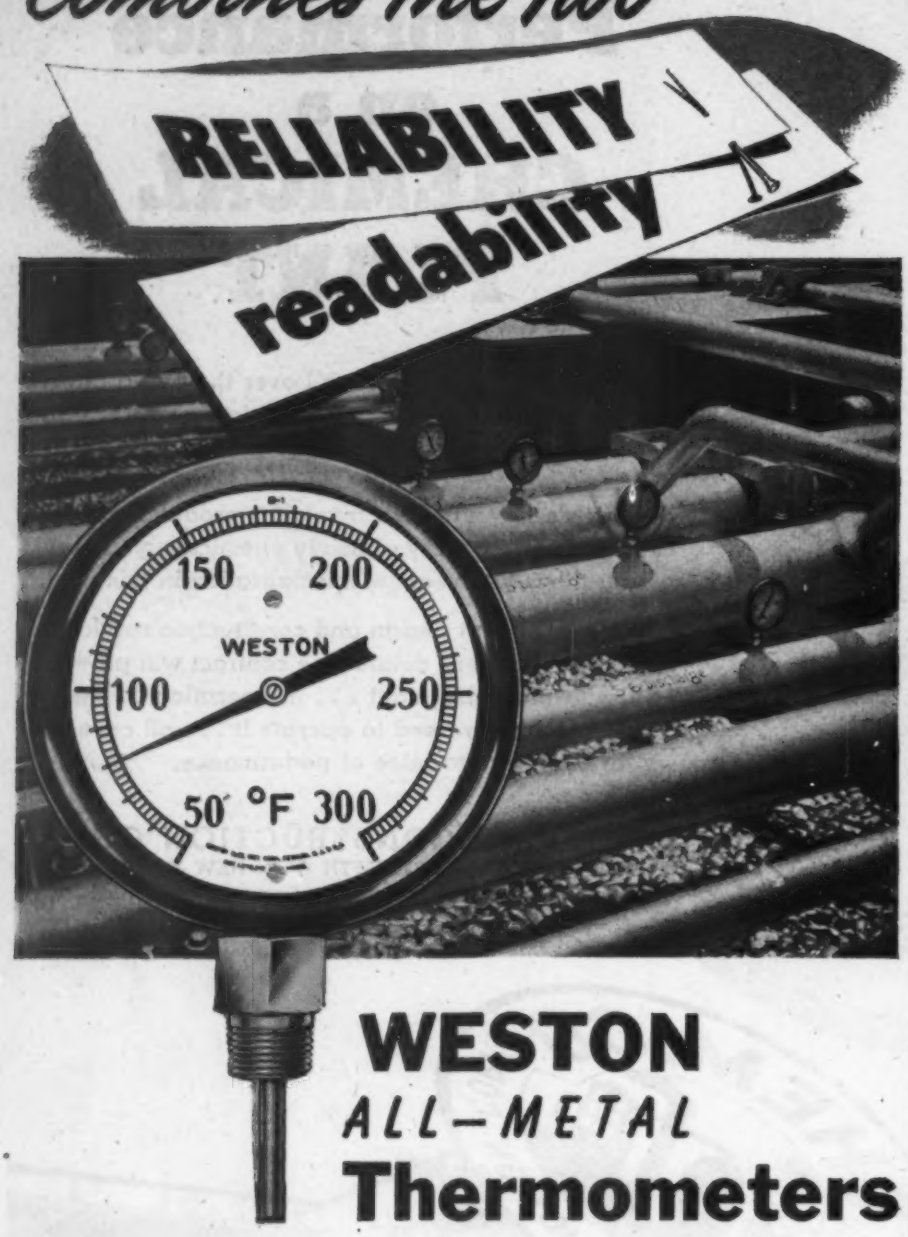

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drocarbons has been used in the development of a method for determining the aromatic hydrocarbon content of aviation gasoline. The authors have modified this method to increase its accuracy, precision, and rapidity. The solubility of the picric acid in gasoline is determined at some temperature between 20° and 30° C. A correction is then applied for temperature to obtain the solubility at 25° C. and the percentage of aromatics is found by reference to a standard curve. The presence of olefins and naphthenes requires that a slight correction be made. This corrections is —1.4 percent for each 10 percent of olefins, and —0.6 percent for each 10 percent of naphthenes. The method was found to be accurate to ±1 percent in the range of 0 to 25 percent of aromatics.

Determination of Hydrogen Sulfide in Gases. EDMUND FIELD AND C. S. OLDACH, *Ind. & Eng. Chem., Anal. Ed.*, **18** (1946) pp. 665-67.

Two methods based on colorimetry were developed for the analysis of traces of hydrogen sulfide in gases. The hydrogen sulfide is first absorbed in caustic solution, and in the more sensitive method is converted to bismuth sulfide. Concentration of the resulting suspension is determined by means of a spectrophotometric measurement. If a spectrophotometer is not available the sulfide is caused to react with a uranylcadmium reagent and an analysis is made by visual comparison in a chromometer. By use of the spectrophotometric method as little as 7 micrograms of hydrogen sulfide can be determined with a precision of ±10 percent. For larger samples the precision improves to ±3 percent. If the chromometer must be used five times as much hydrogen sulfide is required for equal precision, but even then the method is more sensitive than titration.

Conversion of Organic Sulfur to Hydrogen Sulfide for Analysis. EDMUND FIELD AND C. S. OLDACH, *Ind. & Eng. Chem., Anal. Ed.*, **18** (1946) pp. 668-69.

In many modern catalytic processes traces of hydrogen sulfide or organic sulfur compounds in gases or liquids are detrimental to the reaction and it is important to have available methods that permit the quantitative determination of the sulfur compounds. In order to determine organic sulfur compounds they are quantitatively converted to hydrogen sulfide by passing the substance with hydrogen over alumina at 900° C. This technique makes possible the determination of traces of organic sulfur compounds in gases or liquids when the procedure is used in conjunction with a sufficiently sensitive method for analysis of the hydrogen sulfide formed.

Identification of Sulfur Compounds in Gas Mixtures. C. S. OLDACH AND EDMUND FIELD, *Ind. & Eng. Chem., Anal. Ed.*, **18** (1946) pp. 669-72.

A new method was developed for identifying and determining the concentration of organic sulfur compounds when present in concentrations of a few parts per million. This method depends on differences in the solubilities of the sulfur compounds in an inert solvent. The procedure used is based on differences in solubility in an inert solvent used in conjunction with a highly sensi-

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tive analytical method. A complete analysis involves an absorption and a stripping run in a multiple-plate saturator. The total sulfur content of the gas leaving the saturator is plotted against gas volume. A stepwise curve results, such that the gas volume at which the sulfur concentration changes abruptly is characteristic of a specific sulfur compound and the magnitude of the change is a measure of the concentration of that compound. The results of runs made on partially purified manufactured gas and coke-oven gas are given. These are in general agreement with previous industrial experience. An exhaustive evaluation of the method for determining its accuracy has not yet been made.

Determination of Carbon and Hydrogen by Combustion. D. D. TUNNICLIFF, EDWARD D. PETERS, LOUIS LYKKEN, AND F. D. TUEMMLER, *Ind. & Eng. Chem., Anal. Ed.*, **18** (1946) pp. 710-18.

The authors stress that there has been need for a macromethod for determination of carbon and hydrogen that is accurate, precise and rapid under normal operating conditions. A dual-combustion apparatus and improved procedure are described. The dual feature, absence of rubber connections, provisions for adequate combustion control, and well-defined procedure combine to give accurate results in minimum time Other features are control and indication of all gas-flow rates, adequate and convenient pyrometer temperature indication, addition of extra oxygen between the sample and the catalyst, use of carefully determined combustion and operating conditions, and a procedure for accurate analysis of volatile samples. Using the long, precision procedure, a skilled operator can make two determinations in an 8-hour day with a precision of ±0.008 percent hydrogen and ±0.009 percent carbon and a probable accuracy of 0.011 percent hydrogen and 0.015 percent carbon. Using the short, routine procedure, an experienced operator can make eight determinations daily with a probable accuracy of 0.05 percent for each element. Both of the methods are applicable in the presence of sulfur, halogens, and nitrogen.

Book Review

Petroleum Almanac

As the "Petroleum Almanac" the National Industrial Conference Board has published a statistical record of the petroleum industry. The book consists of more than 400 statistical tables, showing the economic significance of the industry, its place in the nation's distributive system, tax revenues and the growing importance of the natural-gas industry.

The content of the book of 420 pages, including index, is divided into 10 major sections, statistical tables and text on production, transportation, refining, marketing, distribution, utilization, finances, labor, regulatory bodies, taxation, alternate sources of energy, prices, value and world petroleum supplies.

The volume is available through National Industrial Conference Board, Inc., 247 Park Avenue, New York 17, N. Y., paper binding, $4.00; cloth binding, $5.00.

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Action of steam in Terry wheel turbine. The steam issues from an expanding nozzle at high velocity and enters the side of the wheel bucket in which its direction is reversed 180°. As this single reversal uses but a portion of the available energy, the steam is caught in a stationary reversing chamber and returned again to the wheel. This process is repeated several times until practically all of the useful energy has been utilized.

r Full Details ask for Bulletin S-116

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Digest of Recently Issued United States

Patents Pertaining to Petroleum Refining

Compiled by HEINZ HEINEMANN

REFINING

U.S.P. 2,408,947 Azeotropic Distillation of Aliphatic Hydrocarbons Having the Formulas C_nH_{2n-2} from Hydrocarbon Mixtures Comprising Same. H. S. Nutting and L. H. Horsley to The Dow Chemical Company.

To a hydrocarbon mixture containing a diolefin with more than 2 and less than 7 C atoms and a more saturated hydrocarbon with the same number of C atoms ammonia is added and the resultant mixture is fractionally distilled at a pressure not below atmospheric pressure. A relatively-low-boiling azeotrope of ammonia and the more saturated hydrocarbon is formed. Butadiene-1, 3 can, for example, be separated by this process from butene-1.

U.S.P. 2,408,999. Motor Fuels. A. E. Robertson to Standard Oil Development Company.

The motor fuel according to the patent comprises 80-95 percent of a branched alkanol of 3-5 C atoms and a minor proportion of a normally gaseous hydrocarbon of 3-5 C atoms in sufficient amount to raise the Reid vapor pressure of the blend to 5-13 psi at 100° F. The gaseous hydrocarbon can be either of aliphatic or of cyclo-aliphatic nature. For example, the motor fuel can comprise 80-95 percent by volume of isopropylalcohol and 20-5 percent of a branched C_4-C_8 paraffinic hydrocarbon, such as isopentane.

U.S.P. 2,409,059. Process for Refining Mineral Oils. J. V. Montgomery, L. B. Goodson and R. W. Henry to Phillips Petroleum Company.

Mineral oil is mixed with a solvent mixture comprising pipecoline, acetone as an anti-solvent, and water. A raffinate phase and an extract phase are formed and are separated. The pipecolines are completely miscible with petroleum oils at much lower temperatures than are the corresponding unsaturated pyridine compounds.

U.S.P. 2,409,156, 2,409,157. Fuel Composition. W. A. Schulze and R. C. Alden to Phillips Petroleum Company.

The fuel supplied to an aviation gasoline engine during an operation period consists of a gasoline comprising essentially a mixture of C_5-C_8 isoparaffins, 2-10 percent by volume of isopropyl benzene, and at least 3 ml. of tetraethyl lead fluid per gallon. The hydrocarbons are so proportioned that said mixture boils in the gasoline range and contains just enough isopentane to give a Reid vapor pressure not greater than 7 pounds. During said operation period rich-mixture operating conditions are maintained in the aviation engine, the fuel- air ratio being at least about .09.

According to U. S. Patent 2,409,157 the isopropyl benzene of said fuel is replaced by a mono-butylbenzene other than normal butylbenzene, e.g. tertiary butlybenzene.

U.S.P. 2,409,167. Motor Fuels. P. L. Veltman to Texaco Development Corporation.

A motor fuel which is particularly adapted for use in super-charged aviation engines, comprises a hydrocarbon-base fuel, such as high-antiknock-base fuel, in which there is dissolved a minor amount of an organo-metallic antiknock agent. This agent essentially comprises a product obtained by heating under pressure an unsaturated hydrocarbon, e.g. an olefin, or a diolefin, and a carbonyl of a metal of atomic number 26-28. Iron, nickel, or cobalt carbonyl may be used. The base fuel can comprise aliphatic and aromatic hydrocarbons.

U.S.P. 2,409,171. Motor Fuels. P. L. De Verter to Standard Oil Development Company.

The motor fuel according to this patent comprises a hydrocarbon mixture composed predominantly of saturated aliphatic hydrocarbons, containing a small amount of a lead alkyl antiknock agent which in the presence of said hydrocarbons tends during storage to form lead-containing precipitates, and hydroquinone or quinhydrone in an amount sufficient to inhibit the formation of a lead-containing precipitate. The hydrocarbon base stock used preferably consists of liquid alkylates boiling within the gasoline-boiling range and manufactured by alkylation of normal olefins with isoparaffins.

U.S.P. 2,409,250. Separation of Butadiene. E. J. Cannon and H. A. Stueve to Carbide and Carbon Chemicals Corporation.

A gas mixture containing butadiene and hydrocarbons of greater volatility than butadiene is scrubbed with a solvent. The solution obtained contains butadiene and some of the more volatile hydrocarbons. This solution is distilled, and a butadiene fraction is removed at a point below the head of the column and above the feeding point. Part of the vapors from the top of the column is condensed and returned as reflux to the column while the uncondensed vapors are returned to the scrubbing operation. Butadiene of high purity is obtained.

U.S.P. 2,409,691. Method for Recovering Hydrocarbons from Gases. F. G. Noble to Standard Oil Development Company.

High-pressure gas is absorbed in lean heavy oil to form a rich heavy oil. Vapors are flash-distilled from the rich oil and contacted with a lean light absorption oil. The residual rich heavy absorption oil is heated and then separated into a vaporous fraction and a liquid fraction. This liquid fraction is passed to a distillation zone to separate absorbed constituents as overhead and a lean heavy absorption oil as bottoms. The rich light absorption oil obtained before is passed to a second distillation zone where absorbed constituents are separated as overhead and a lean light absorption oil is obtained as bottoms. Vapors separated in the heating of the rich heavy absorption oil are passed to the second distillation zone to aid in stripping absorbed constituents from the light absorption oil.

U.S.P. 2,409,746. Motor Fuels. T. W. Evans and A. G. Cattaneo to Shell Development Company.

The motor fuel for supercharged spark-ignition engines described in this patent comprises a mixture of methyl tertiarybutyl ether and gasoline hydrocrabons containing isooctane, neohexane, or triptane, or mixtures thereof. The fuel mixture has an octane rating lower than that of said hydrocarbons and an A.B. ratio greater than that of said gasoline hydrocarbons.

U.S.P. 2,410,042. Purification of Styrene and Its Homologues. G. R. Bond, Jr., to Houdry Process Corporation.

Hydrocarbon oil produced by cracking a gas oil and containing a styrene compound is treated by dissolving SO_2 in the oil and maintaining the resulting solution at a temperature below 130° F. for at least 15 minutes in the absence of undissolved liquid-phase SO_2. Reactive impurities present are converted to a high-boiling reaction product which is separated from the hydrocarbon oil.

U.S.P. 2,410,166. Process of Separating Toluene. T. B. Kimball to Sinclair Refining Company.

Toluene is separated from its mixture with paraffin of similar boiling point by intimately contacting the mixture with liquid SO_2. A raffinate phase and a primary extract phase are formed. The extract phase containing the SO_2 is intimately contacted with a paraffin hydrocarbon having a boiling point substantially higher than that of toluene. Two component parts are formed, viz. a washed extract and a spend wash oil. Hydrocarbons with boiling point similar to toluene are separated from the spent wash oil by distillation, while toluene is recovered from the washed extract by distillation.

U.S.P. 2,410.223. Treatment of Hydrocarbons. H. R. Legatzki to Phillips Petroleum Company.

A low-boiling fraction of impure hydrocarbons is fractionated and purified by introducing said hydrocarbon fraction into a fractionating column intermediate its ends, removing overhead the moisture contained in the hydrocarbons and a portion of chemically-reactive impurities, such as hydrogen sulphide, and withdrawing at least a portion of the partially purified hydrocarbon fraction at a point slightly below that of introduction of the hydrocarbons. The withdrawn hydrocarbons are treated with a chemical inert to these hydrocarbons for removal of chemically-reactive impurities still contained therein. The

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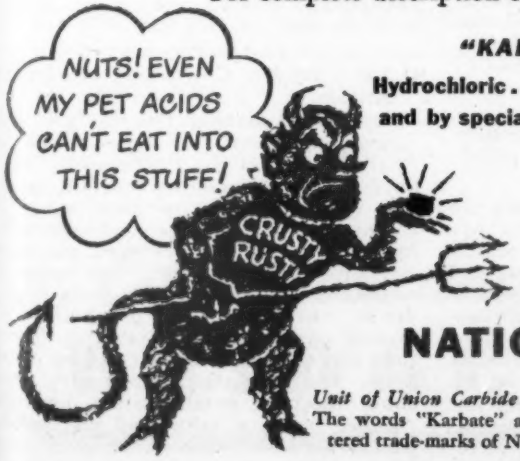

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chemical and the reaction products are then separated, and the treated hydrocarbon fraction is reintroduced into the fractionator at a point immediately below its point of withdrawal and a substantial distance above the bottom of the fractionator. The removal of moisture and of the chemically-reactive impurity is completed by continual fractionation. The purified and dehydrated hydrocarbon fraction is removed from the bottom portion of the fractionator. If the impurity to be removed is hydrogen sulfide, a caustic material is used for the treatment of the hydrocarbons; if the impurity, however, consists of unsaturated hydrocarbons, the purification is carried out by means of an acid, such as sulfuric acid.

U.S.P. 2,410,356. Purification of Tetraethyl Lead. A. E. Parmelee to E. I. du Pont de Nemours & Company.

Steam-distilled tetraethyl lead containing sludge-forming impurities is purified by washing it with at least one-fifth of its volume of an aqueous solution of hydrogen peroxide of 0.35-5 per cent concentration.

U.S.P. 2,410,496. Separation and Recovery of Unsaturated Gases. R. A. Graff to The Girdler Corporation.

Butadiene is separated from a solution containing a dissolved cuprous-cupric ammonium acetate solution, as used for the selective separation of unsaturated gases from mixtures, by extracting the butadiene from the said combined solution by a stripping agent which is immiscible with this solution, such as a liquid hydrocarbon having a boiling point substantially above that of butadiene. The stripping agent is then separated from the butadiene in a desorber by blowing with a gaseous medium, such as ammonia. The butadiene vapours formed are then condensed, and a part of the liquid butadiene is returned to the top of the desorber as a reflux.

U.S.P. 2,410,583. Separation of Hydrate-Forming Components of Gaseous Mixtures. A. J. L. Hutchinson to The Fluor Corporation, Ltd.

A gas comprising a mixture of hydrate-forming hydrocarbon components and an inert non-hydrate-forming constituent is contacted with water and a hydrate-forming halogenated aliphatic hydrocarbon additive, such as CCl_4, in a conversion zone under conditions of temperature and pressure such as to cause the formation of a mixed hydrate of the two hydrateable compounds. This mixed hydrate has substantially lower vapor pressure than the hydrates of the individual hydrocarbon components at the corresponding temperature. The inert constituent is separated from the mixed hydrate, the mixed hydrate is continuously removed from the conversion zone and dissociated. The additive is recovered and returned to the conversion zone.

CATALYST PREPARATION ACTIVATION AND REGENERATION

U.S.P. 2,408,986. Gel Bead. M. M. Marisic and A. C. Schmitt to Socony-Vacuum Oil Company.

A hydrosol of inorganic oxide including silicia and a metal oxide, such as Al_2O_3, characterized by an inherent capacity to set a hydrogel after a suitable period of time without addition to or subtraction from the sol of any substance, is treated by dispersing in the sol particles of a combustible solid of less than 300-mesh size. The sol is then admitted in the form of separate globules to a body of liquid medium immiscible with water in which the globules assume spheroidal shape due to surface tension at the interface between the sol and the liquid medium. The spheroidal globules are retained in this medium until gelatin occurs and are then dried. Thereafter the combustible solid is burned by contacting the dried gel with an oxidizing gas at combustion temperature. Lamp black may be used as the combustible solid which serves to increase the porosity of the gel beads formed.

U.S.P. 2,408,996. Catalyst Rejuvenation. R. L. Parker and H. C. Huffman to Union Oil Company of California.

The carbon-hydrogen ratio of hydrocarbon oils is changed by heating the hydrocarbons to reaction temperature, e.g. to 200-600° C., in the presence of a catalyst for a period of time during which the catalyst becomes coated with a carbonaceous deposit. The spent catalyst is regenerated by removing the carbonaceous deposit at a temperature not more than about 100° C. above the reaction temperature. This cycle of operations is repeated until the catalyst's activity after regeneration is substantially lower than its original activity. The catalyst is then rejuvenated by heating it to a temperature of 700-900° C. for a period of between 1 minute and 10 hours. Thereafter the catalyst is cooled to the reaction temperature at a rate not in excess of about 5° C. per minute. An alumina catalyst can be used, and its rejuvenation can be carried out in the presence of air.

U.S.P. 2,409,234. Revivifying Adsorptive Material. M. H. Arveson to Standard Oil Company (Indiana).

Solid contacting material contaminated with adsorbed hydrocarbon matter from the decolorization of oils, e.g. mineral oils, is dispersed in a stream of air and passed upwardly through a burning zone. A portion of the contacting material settles while the major part is maintained within the burning zone in a turbulent mass having a density of 5-30 pounds per cubic foot. The temperature maintained throughout the burning zone is sufficient to remove substantially all of the adsorbed hydrocarbon matter. Additional quantities of gases are introduced into the burning zone at vertically spaced points. Revivified contacting material is continuously withdrawn overhead together with the stream of gases. The withdrawn mixture is cooled, the revivified contacting material is recovered from the gases and accumulated in an aerated dense phase.

U.S.P. 2,409,494. Catalyst Manufacture. G. H. Keating to The Texas Company.

An inorganic catalyst gel, such as silicia gel, is mixed in moist and hydrated state with an amount of a starch material corresponding to 30-60 percent of the dry weight of the catalyst gel. The plastic mass is extruded in substantially tubular form. The extruded catalyst is cut into rings which are dried at a temperature of 150-300° F. in an atmosphere maintained at about 20-50 per cent relative humidity, prior to calcination.

U.S.P. 2,410,436. Catalyst and Catalytic Processes. F. J. Ewing to Filtrol Corporation.

A cracking catalyst is described which comprises an acid activated sub-bentonite montmorillonite clay impregnated with hydrated alumina. The efficiency of the catalyst is in excess of four times that of the native clay employed in the production. The hydrated alumina used in the impregation is formed by the treatment of an aluminum salt with a base.

U.S.P. 2,410,558. Manufacture of Catalyst. G. M. Webb and M. A. Smith to Universal Oil Products Company.

Precipitated and purified hydrated alumina is mixed with 1-10 percent by weight of an ammonium salt, such as NH_4Cl. The mixture is dispersed in an aqueous medium, such as acidulated water, to form an alumna hydrosol. A metal compound soluble in this aqueous medium and capable of yielding a catalytically-active metal oxide upon heating, particularly a compound of a metal from the left column of group VI of the periodic table, such as a chromium or a molybdenum compound, is added to said hydrosol. The resultant mixture is dried, and the dried material is heated to convert the metal compound to the metal oxide. The catalysts obtained have a superior activity for example in the reforming of low antiknock gasoline and naphthas.

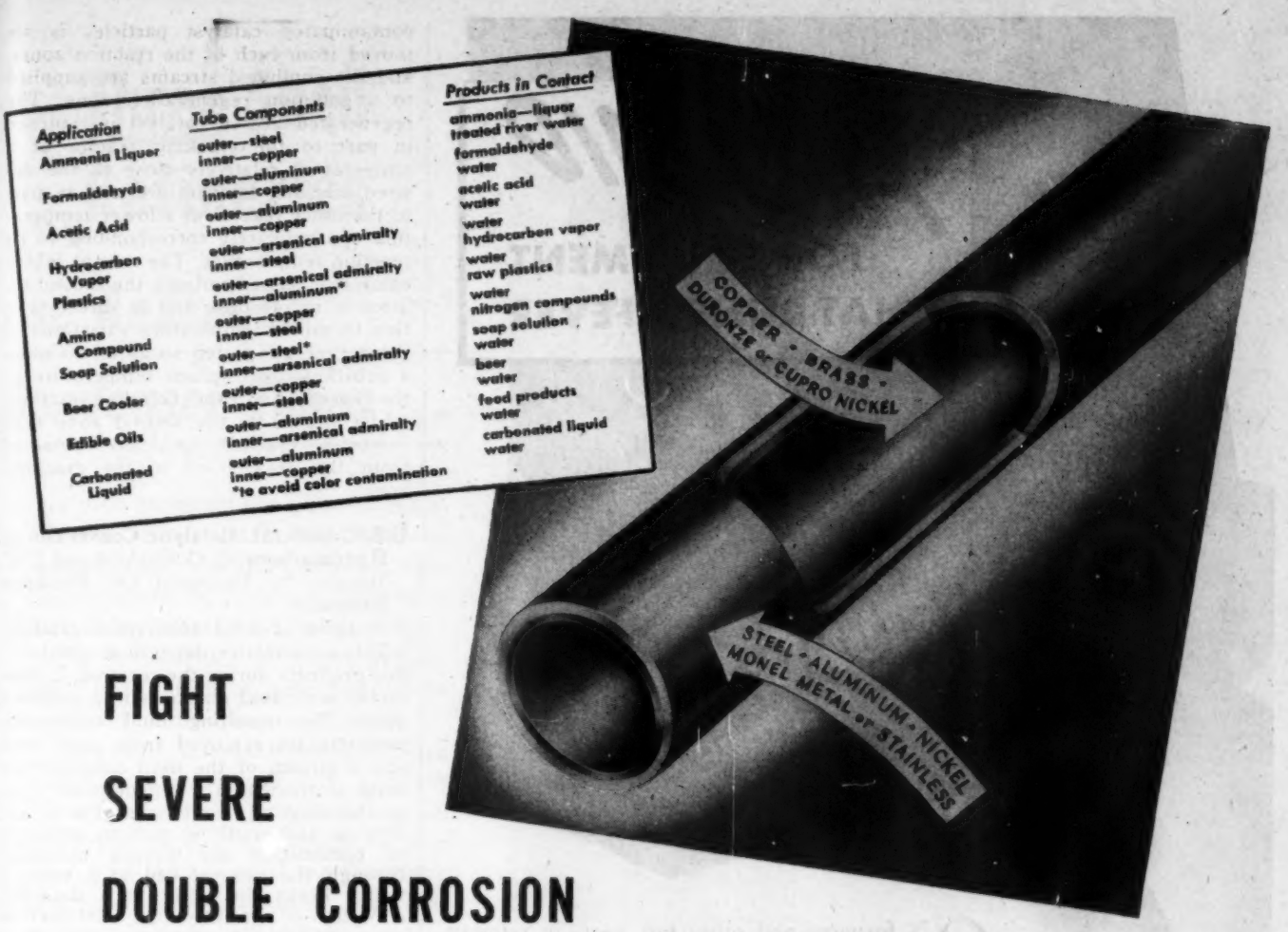
CRACKING AND REFORMING

U.S.P. 2,408,948. Catalytic Hydrocarbon Reforming Process. C. L. Ocon and E. A. Ocon, the former assignor to the latter.

Hydrocarbons of gasoline-and naphtha-boiling range are reformed to high-antiknock motor fuel by heating the charge stock jointly with a hydrogen-bearing gas containing about 50 mol-percent hydrogen to a temperature of 925-1050° F. under superatmospheric pressure. The heated mixture is subjected to contact with a reforming catalyst and is then passed to a high-temperature reheating zone wherein its temperature is rapidly raised to at least the average reaction temperature. The reheated mixture is again contacted with a reforming catalyst, withdrawn, cooled and passed to a flash zone for separation of gas from condensate. The condensate is passed to a zone where hydrocarbons with not more than 4 C atoms are separated. A portion of the heavier hydrocarbons is recycled for further heating and catalytic reformiing jointly with the reaction mixture.

U.S.P. 2,409,353. Conversion of Hydrocarbons. C. J. Giuliani and C. H. Angell to Universal Oil Products Co.

Separate streams of active cracking catalyst particles are continuously supplied to separate confined reaction zones. The catalyst in one of these zones is contacted under cracking conditions with hydrocarbon oil heavier than gasoline, and the catalyst in the other zone is contacted with gasoline fractions in order to convert them to a product of improved quality at a temperature lower than that employed in the cracking operation. The converted gasoline fractions show improved octane rating, bromine number, blending value, and susceptibility to antiknock agents. A stream of



Application	Tube Components	Products in Contact
Ammonia Liquor	outer—steel inner—copper	ammonia—liquor treated river water
Formaldehyde	outer—aluminum inner—copper	formaldehyde water
Acetic Acid	outer—aluminum inner—copper	acetic acid water
Hydrocarbon Vapor	outer—arsenical admiralty inner—steel	water hydrocarbon vapor
Plastics	outer—arsenical admiralty inner—aluminum*	water raw plastics
Amino Compound	outer—copper inner—steel	water nitrogen compounds
Soap Solution	outer—steel* inner—arsenical admiralty	soap solution water
Beer Cooler	outer—copper inner—steel	beer water
Edible Oils	outer—aluminum inner—arsenical admiralty	food products water
Carbonated Liquid	outer—aluminum inner—copper *to avoid color contamination	carbonated liquid water

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contaminated catalyst particles is removed from each of the reaction zones, and the combined streams are supplied to a common regeneration zone. The regenerated heated catalyst is returned in part to the cracking reactor at a temperature relatively close to the desired cracking temperature, and in part to the other reactor at a lower temperature approximately corresponding to its reaction temperature. The rate at which catalyst is passed through the second reactor is in response and in direct relation to minor temperature variations in the regenerating step so as to maintain a substantially constant temperature in the regeneration zone. Gasoline fractions to be treated in the second zone may comprise cracked products obtained from the heavier oil in the cracking zone.

U.S.P. 2,409,751. Catalytic Conversion of Hydrocarbons. C. G. Gerhold and J. E. Burgess to Universal Oil Products Company.

A mass of solid subdivided catalyst, which accumulates deleterious combustible products during the reaction, is contacted with fluid reactants in a confined zone. The resulting fluid conversion products are removed from said mass and a stream of the used solid-particle mass is treated in a regeneration zone by burning off combustibles with air. The air and resulting gaseous products of combustion are passed upwardly through the catalyst bed at a velocity which keeps the latter in a fluid-like condition of relatively high solid-particle concentration, maintaining a light-phase region of materially reduced solid-particle concentration above the bed. A stream of solid particles from said bed is returned to the reaction zone without passing it through said light phase, while gaesous products from the regeneration zone are discharged through the light phase. A least a portion of the gaseous products is burned outside said light-phase region and noncombustible diluent gas is supplied to this region in an amount dependent on the temperature of the last-mentioned combustion step. The combustible content of the gaseous products in the light phase is thus kept low enough to make the mixture in this phase non-inflammable.

U.S.P. 2,410,284. Catalytic Cracking System. R. C. Gunness and J. W. Jewell to Standard Oil Company (Indiana), and The M. W. Kellogg Company.

The fluid-type catalytic-cracking system according to this patent comprises a gasoil surge drum, a regeneration system containing a heat exchanger, a fractionating system containing a heat exchanger, a steam-generation system, means for passing gasoil from the surge drum to the heat exchanger in the regeneration system, means for passing hot gas from this heat exchanger to the heat exchanger of the fractionation system, means for passing gasoil from the heat exchanger of the regeneration system to the steam generator, and means for passing gasoil from the heat exchanger of the fractionation system and steam generator, respectively, back to the gas surge drum. The spent catalyst is passed from the conversion system of the plant to the regeneration system, and the regenerated catalyst is re-introduced to the conversion system, where

Three Shell Refineries use Anaconda Condenser Tubes...

AT THE WOOD RIVER, Illinois; Houston, Texas; and Norco, Louisiana refineries of Shell Oil Company, sizable quantities of Anaconda Arsenical Admiralty Tubes have been installed.

The accompanying photographs show installation of tube bundles so equipped at the Houston refinery.

Anaconda Arsenical Admiralty is a widely used condenser tube alloy in the refinery field. Pioneer in the development and manufacture of this tube, The American Brass Company also produces eleven other standard and several special alloys for condenser and heat exchanger tubes.

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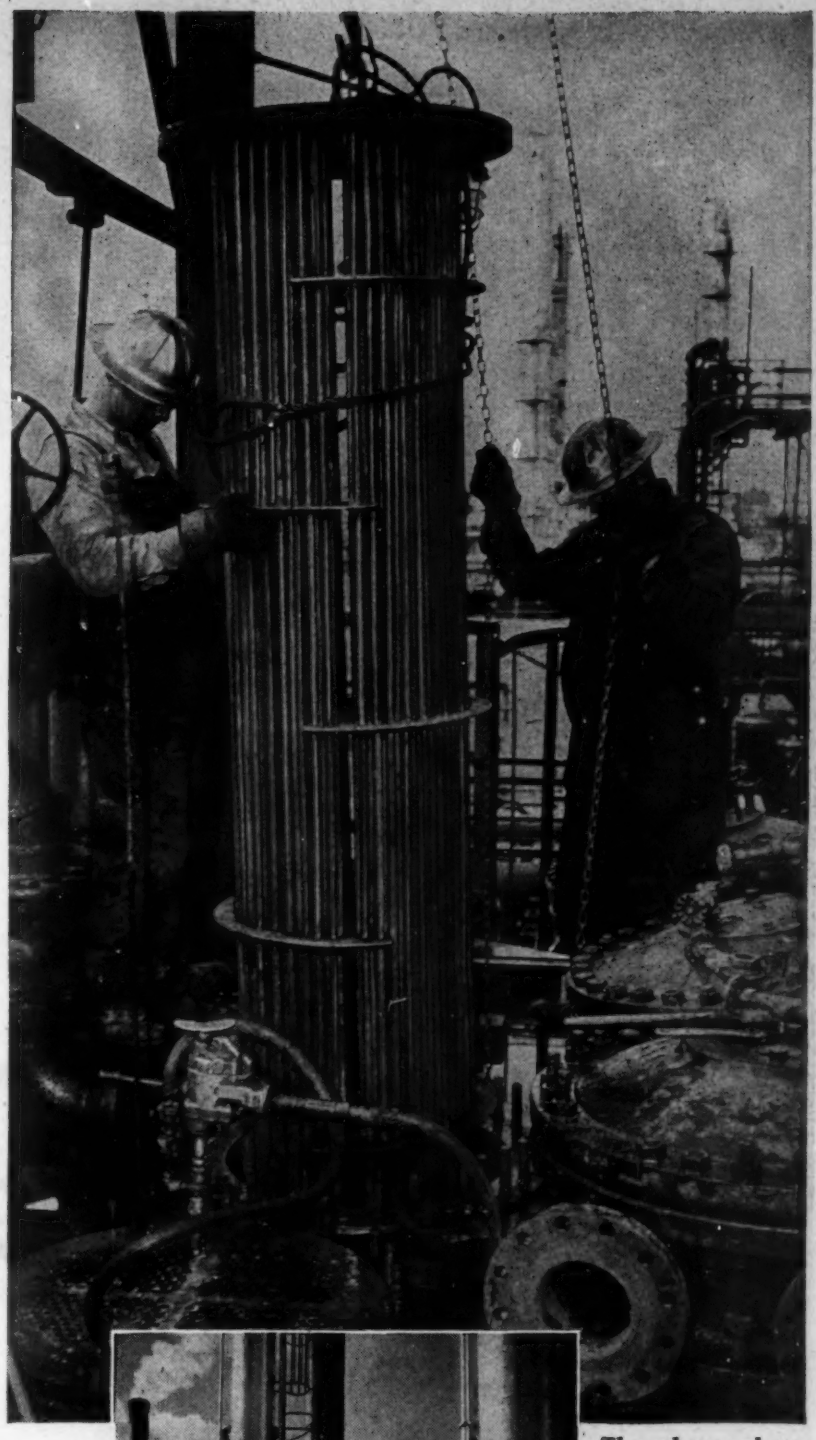
Equipment manufacturers are invited to utilize the broad experience and metallurgical knowledge of our Technical Department in determining the most suitable alloys. Publication B-2 provides comprehensive information on Anaconda Condenser materials. It will be mailed on request. 492TS



The above photograph shows installation of tube bundle equipped with Anaconda Condenser Tubes at the Houston, Texas refinery of Shell Oil Company. At left is view of one of refinery units.

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Guy Maher

Guy Maher, Plant Engineer
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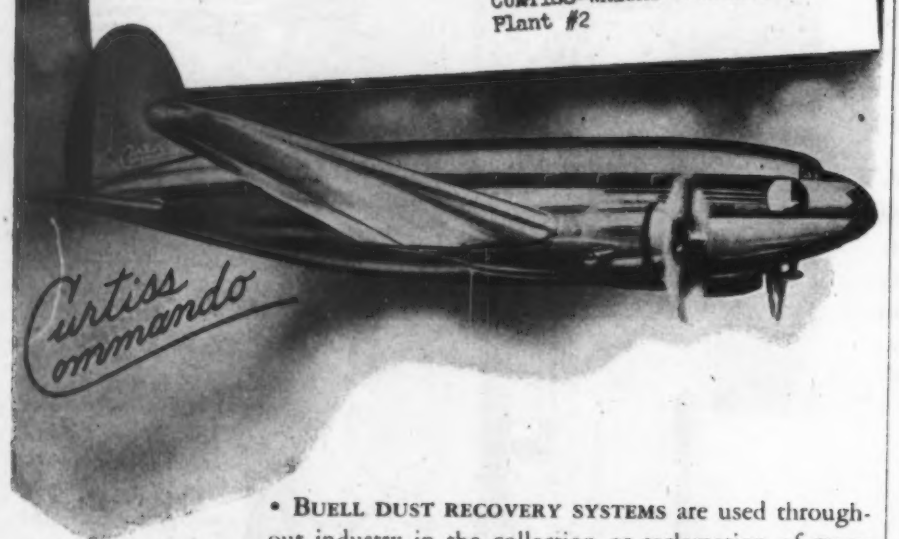

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part of the preheated gasoil is converted.

U.S.P. 2,410,309. Continuous Catalytic System. Th. P. Simpson, J. W. Payne, and J. A. Crowley, Jr., to Socony-Vacuum Oil Company.

Heavier petroleum hydrocarbons are continuously cracked in the presence of a particle-form catalyst, which is moved downward as a substantially compact column of particles through a conversion zone operating under pressure while vapors of the hydrocarbons to be converted are passed through this catalyst column under cracking conditions. Active catalyst particles are introduced near the top of the conversion zone, while spent particles are withdrawn near its bottom, so that a column of active catalyst is maintained. In order to prevent the escape of gaseous hydrocarbons the compact catalyst stream is extended above and below the catalyst column to such an extent that the head of catalyst created thereby is greater than the pressure in the conversion zone. The catalyst thus feeds by gravity from the catalyst stream to the column of catalyst without passing through any vapor-sealing catalyst valve and the spent catalyst is withdrawn at the lower end of the catalyst stream through a zone of restricted cross-section such that here, too, substantial leakage of gaseous hydrocarbons is prevented without the use of a vapor-sealing catalyst valve.

U.S.P. 2,410,316. Production of Motor Fuel. C. L. Thomas to Universal Oil Products Company.

High-octane motor fuel is produced by thermally cracking hydrocarbon oil boiling above the range of gasoline at a temperature of 900-1050° F. and at a pressure of 15-150 psi if the temperature is below 975° F., or at a pressure of 15-250 psi if the temperature is higher. Not more than 20 percent by weight of the oil shall be cracked per pass. A gasoline fraction is separated from the cracking products and is subjected to the action of an olefin isomerizing catalyst at a temperature of 800-1100° F. and at mildly superatmospheric pressure for a time of contact equivalent to an hourly liquid space velocity of 5-50 volumes of gasoline per hour per volume of catalyst when the reacting temperature is 950° F.

ISOMERIZATION

U.S.P. 2,408,950. Treatment of Hydrocarbons. H. Pines and H. S. Bloch to Universal Oil Products Company.

Hydrocarbons are converted in the presence of a hydrogen halide. A gas mixture containing hydrogen halide and normally gaseous hydrocarbons is separated from the reaction products. This gas mixture is contacted with a solid adsorbent which will adsorb hydrogen halide. Thereafter the solid adsorbent is contacted in liquid phase with at least a portion of the hydrocarbon to be treated, dissolving adsorbed hydrogen halide in the liquid. The resultant hydrogen halide-containing hydrocarbons are charged to the reaction zone. The process can be used for converting normal butane to isobutane, or for alkylating an isoparaffin with an olefin.

U.S.P. 2,409,260. Butane Isomerization. E. L. d'Ouville and B. L. Evering to Standard Oil Company (Indiana).

A charge stock consisting chiefly of

n-butane is continuously passed in liquid phase through a column of liquid $AlCl_3$-hydrocarbon complex in the presence of HCl while maintaining isomerizing conditions. Said complex is produced by reacting $AlCl_3$ in the presence of HCl with a saturated hydrocarbon free from olefins and aromatics. The amount of reactants employed in producing the complex is such that the complex will have a hydrocarbon content within the range of 16-40 percent by weight, based on total complex. Catalyst activity is maintained by adding $AlCl_3$ to the complex.

U.S.P. 2,410,024. Production of Isobutane and the Like Hydrocarbons. E. W. M. Fawcett and J. H. Beynon to Anglo-Iranian Oil Company, Ltd.

Normal aliphatic hydrocarbons are converted into the corresponding branched-chain hydrocarbons by contacting an aliphatic feed stock with an aluminum-halide catalyst in the presence of tetra-chlorethane at elevated temperature and under sufficient pressure to maintain the solution in the liquid phase. In the isomerization of n-butane at temperature of about 100° C. is employed, and the amounts of catalyst and tetra-chlorethane used correspond to 10 percent and 50 percent, respectively, by weight of the butane.

ALKYLATION

U.S.P. 2,409,080. Preparation of Alkylated Cyclic Hydrocarbons. C. M. Thacker and R. T. Bell to The Pure Oil Company.

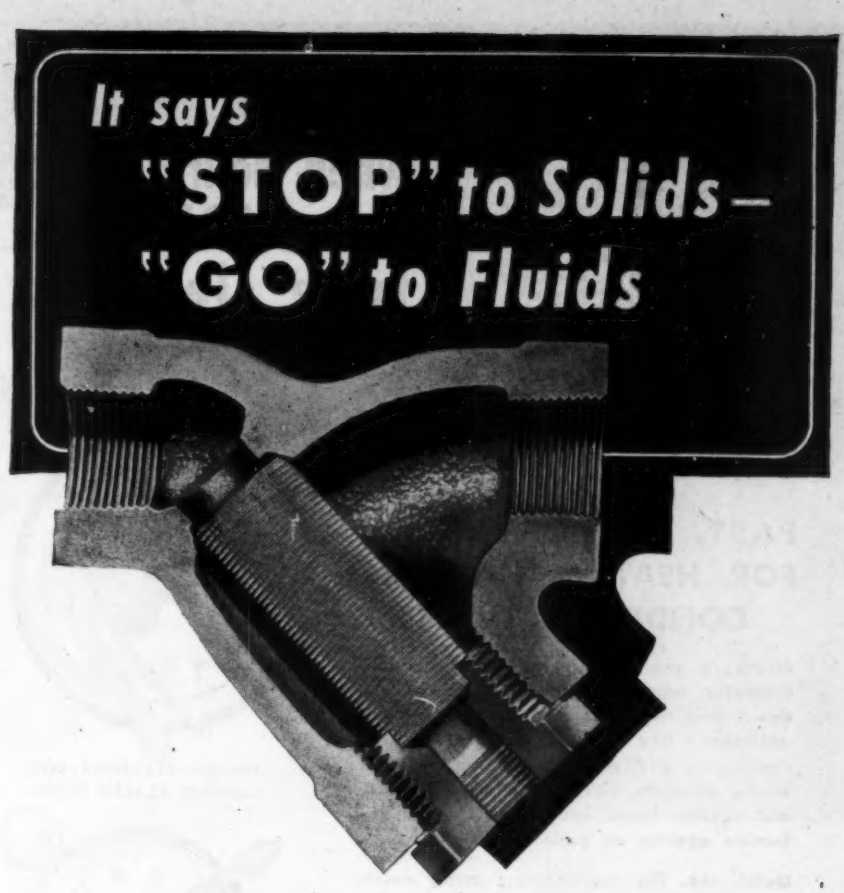
Carbocyclic compounds susceptible to alkylation, such as benzene, are alkylated with alkyl mercaptan or alkyl sulfide in the presence of copper pyrophosphate at a temperature of 200-525° C. The inorganic catalyst can be supported on activated charcoal, and elevated pressure, such as 100-500 psi, may be employed in the alkylation reaction.

U.S.P. 2,409,090. Ethylene Alkylation. R. E. Woodward, W. P. Hawthorne, and J. R. Meadow to Socony-Vacuum Oil Company.

Isoparaffins, such as isobutane, are alkylated with ethylene by dispersing their vapor mixture heated to 400-1000° F. in a body of liquid HF maintained at a temperature of 100-200° F., under sufficient pressure to maintain liquid HF in said body.

U.S.P. 2,409,389. Alkylation Process. C. R. Ringham to Phillips Petroleum Company.

An alkylating reactant, such as ethylene, and an alkylatable hydrocarbon, such as isobutane, are reacted in the presence of a liquid alkylation catalyst, such as $AlCl_3$-hydrocarbon complex. The reaction is conducted in at least two series of reactors having two reactors in each set. A mixture of an excess of alkylatable hydrocarbon and alkylating reactant is passed to the bottom of each of the reactors, and a mixture of alkylatable hydrocarbon and alkylating reactant is also passed to an intermediate point of the first reactor of each set. A portion of the liquid catalyst is passed from a common source to the bottom of the first reactor of each set in an amount not greater than that which will permit the catalyst to be the dispersed phase in the resulting mixture. An intimate hydrocarbon-catalyst mixture is passed from the top of the first reactor to each



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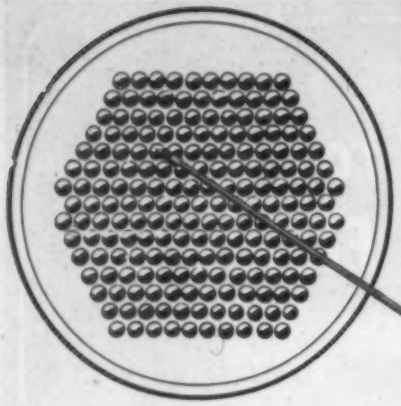

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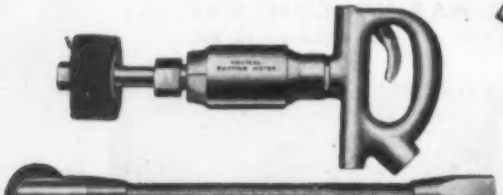

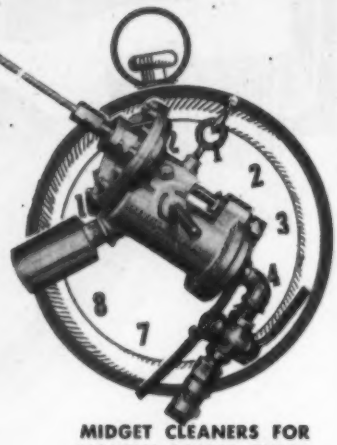

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set to the bottom of the second reactor of the same set. A vigorous and intimate mixing of the contents of each reactor is effected during passage from bottom to top of the reactor. The effluent of each second reactor is passed to a primary settling zone to separate the bulk of the liquid catalyst. The separated hydrocarbon effluent of each primary settling zone is combined as is the catalyst effluent of each of these zones. A cooled portion of the combined hydrocarbon effluent is passed to the bottom of the first reactor of each set, while a further portion thereof is passed to a secondary settling zone where substantially all of the entrained catalyst is removed from the hydrocarbons. The separated catalyst is mixed with the aforesaid combined catalyst effluent to produce a common catalyst source wherein the catalyst is adjusted to maintain a desired catalytic activity by withdrawal of a portion of this catalyst source as spent catalyst and by addition of fresh catalyst constituents.

U.S.P. 2,409,544. Alkylation of Hydrocarbons. L. A. Clarke to The Texas Company.

An aviation gasoline is produced by alkylating a molar excess of isobutane with a cracked C_3-gas fraction containing ethylene and not more than 10-20 percent by weight of propylene based on the ethylene in the presence of an $AlCl_3$ cataylst. The volume ratio of catalyst-to-hydrocarbons used is in excess of .06:1. A temperature of 105-130° F. and a pressure sufficient to maintain isobutane in the liquid phase are maintained in the reaction zone. A promoter is added in an amount sufficient to provide a HCl concentration of less than 0.2 per cent by weight of the hydrocarbon charge. The contact time required for producing a C_8 alkylate containing a major proportion by volume of 2,3-dimethylbutane is 5-20 minutes. In another zone isobutane is alkylated with a C_4 cracked-gas fraction containing butylenees in the presence of a catalyst other than a metal halide to form a C_8 alkylate containing a high proportion of octanes. Gases lighter than C_4 are separated from the C_8 alkylate. The C_8 alkylate and C_4 alkylate are debutanized and blended. Off gases from the debutanizers are combined and fractionated to separate an isobutane-rich fraction from a normal-butane fraction.

U.S.P. 2,409,681. Chemical Process. R. M. Hill and C. H. Watkins to Standard Oil Development Company.

A catalyst adapted to promote and catalyze the reaction between an isoparaffin and an olefin, e.g., between isobutane and ethylene, is prepared by contacting $AlCl_3$ with an isoparaffin and a lower olefin, such as those mentioned above, and an alkyl chloride or HCl at elevated temperatures and pressures for a period of 5½ hours. The catalyst obtained is a brown mobile liquid with a specific gravity of about 1.2.

U.S.P. 2,409,802. Production of Ethylbenzene. L. Schmerling to Universal Oil Products Company.

Benzene, ethyl alcohol and ethylene are reacted at a temperature of 500-750° F. under a pressure of 200-1200 psi in the presence of a solid phosphoric acid catalyst. A ratio of 2-20 mol proportions of benzene to 1 mol proportion of ethyl alcohol and ethylene is maintained. The ethyl alcohol is dehydrated to water

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and ethylene and the benzene is alkylated by ethylene. The reaction products are separated into alkylated benzene and unreacted ethylene and benzene. After recovery of the alkylated benzene the unreacted mixture of ethylene and benzene is cooled under a pressure of 200-1200 psi in order to dissolve unreacted ethylene in the benzene. This solution is recycled to the reaction zone for further conversion.

U.S.P. 2,410,070. Catalytic Alkylation Process. A. W. Horton, to Socony-Vacuum Oil Company.

A paraffinic hydrocarbon and an olefinic hydrocarbon are contacted in gaseous phase under alkylating conditions with a homogeneous gaseous-phase alkylation catalyst consisting essentially of a bromine derivative of a light aliphatic hydrocarbon, in the presence of oxygen. An excess of paraffinic hydrocarbon, such as isobutane, over olefinic hydrocarbon, such as propylene, is employed. The temperature may vary between 590-850° F., and pressures in excess of 500 psi may be used.

U.S.P. 2,410,071. Catalytic Alkylation Process. A. W. Horton, J. W. Brooks, and A. A. O'Kelly to Socony-Vacuum Oil Company.

The process of U.S.P. 2,410,070 is modified by employing an alkylation catalyst consisting essentially of chlorine or bromine. No oxygen need be present in this case. The quantity of halogen employed varies between 1-1.25 percent of the charge.

U.S.P. 2,410,072. Alkylation of Isobutane with Propylene. A. W. Horton, J. W. Brooks, and A. A. O'Kelly to Socony-Vacuum Oil Company.

Isobutane is contacted with propylene, in gaseous phase, at a temperature of 750-850° F. and under a pressure in excess of 2500 psi in the presence of an alkylation catalyst consisting essentially of a chlorine or a bromine derivative of an acyclic hydrocarbon which under alkylation conditions forms a single, homogeneous gaseous phase with the isobutane and the propylene. A chlorinated or brominated naphtha, or isobutylene dibromide are examples of suitable catalysts.

U.S.P. 2,410,107. Alkylation Process. A. N. Sachanen, A. A. O'Kelly, and C. G. Myers to Socony-Vacuum Oil Company.

The process of this invention is similar to that of U.S.P. 2,410,072. The same type of catalyst is used in the alkylation of a paraffinic hydrocarbon with an olefin, in gaseous phase, e.g., in the alkylation of isobutane with ethylene, at a temperature of 590-850° F. and under a pressure in excess of 500 psi.

U.S.P. 2,410,108. Alkylation. A. N. Sachanen, A. A. O'Kelly, and C. G. Myers to Socony-Vacuum Oil Company.

In a process similar to the processes described in U.S.P. 2,410,072 and 2,410,-107, acyclic, alicyclic, or aromatic organic halogen compounds which under the alkylation conditions employed form a single, homogeneous gaseous phase with the normally gaseous paraffinic hydrocarbons and olefins to be treated, are used as catalysts. These catalysts are used in amounts of at least 0.5 percent by weight based on the total weight of the normally gaseous hydrocarbon mixture to be treated.

U.S.P. 2,410,111. Treatment of Hydrocarbons. C. L. Thomas and V. Haensel to Universal Oil Products Company.

Alkylated aromatics are produced by reacting an aromatic hydrocarbon with an olefin in the presence of a silica-zirconia catalyst, e.g., a calcined mixture of the hydrogels of silica and zirconia. Alumina can also be present in the catalyst. An alkylation temperature of 350-850° F. is employed.

U.S.P. 2,410,151. Alkylation of Hydrocarbons. J. D. Danforth to Universal Oil Products Company.

An aromatic hydrocarbon is alkylated with an olefin in a reaction zone containing a solid packing material. $AlCl_3$ vapors are introduced to this zone which is maintained at an alkylating temperature low enough to condense at least a portion of the $AlCl_3$ vapors on the packing material. This temperature can vary between 30° and 300° F. A carrier gas, such as hydrogen or HCl may be introduced along with the $AlCl_3$ vapors.

U.S.P. 2,410,498. Alkylation Process. H. J. Hepp to Phillips Petroleum Company.

A low-boiling alkylatable hydrocarbon (such as isobutane) and ethylene are passed in a mol ration of between 3:1 and 10:1 to a first reaction zone where their mixture is contacted with an aluminum-halide alkylation catalyst under such conditions that the concentration of unreacted ethylene in the effluent is between 0.2 and 3 mol percent of the hydrocarbons present. A high-boiling fraction comprising alkylation products and a low-boiling fraction comprising unreacted ethylene and alkylatable hydrocarbon are separated from the effluent. The low-boiling fraction is passed to a second reaction zone and again subjected to alkylation under corresponding conditions to effect additional formation of alkylate. The alkyl derivatives formed, such as diisopropyl, are recovered from the effluent of the second reaction zone and from said high-boiling fraction.

POLYMERIZATION, CONVERSION

U.S.P. 2,409,247. Polymerization Process. J. W. Brooks, A. A. O'Kelly, and R. H. Work to Socony-Vacuum Company.

Anhydrous HF is reacted with P_2O_5 to form a catalyst reaction mixture. Olefin hydrocarbons are contacted in a separate zone under polymerizing conditions with the catalyst reaction mixture thus obtained. A temperature of 0.60° C. is employed. High-octane motor fuel is produced.

U.S.P. 2,409,248. Polymerization. J. W. Brooks, A. A. O'Kelly, and R. H. Work to Socony-Vacuum Oil Company.

Normally gaseous olefins are contacted with monofluophosphoric acid or difluophosphoric acid in the presence of paraffinic hydrocarbons at temperatures between 32-140° F. and under a pressure sufficient to maintain the olefins in liquid phase. High-octane motor fuel is obtained. Propane, butane, pentane, isobutane and isopentane can be used as the paraffinic hydrocarbons. The catalytic material is prepared outside the polymerization zone.

U.S.P. 2,409,727. Production of Olefin Polymers. G. C. Bailey to Phillips Petroleum Company.

Di-isobutylene is contacted with P_2O_5

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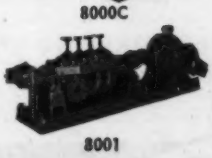
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at a temperature of 0-100° C. for a period of time such that an optimum amount of tetra-isobutylene is produced. The treating period will preferably be between 3-7 hours.

U.S.P. 2,409,996. Ethylene Polymerization. M. J. Roedel to E. I. du Pont de Nemours & Company.

The polymerization of ethylene at a temperature of 100-400° C. and under a pressure of 400-3000 atmospheres is carried out in the presence of an alkyl hydroperoxide, such as tertiary butyl hydroperoxide. Preferably 0.0005-2 percent of such a hydroperoxide based on the total weight of ethylene is employed. An inert, normally liquid organic medium, such as benzene, can be added to the hydroperoxide catalyst. The use of this type of catalysts yields high mol weight, tough, high-softening ethylene polymers free of inorganic contaminants and possessing excellent solubility and melt extrusion characteristics.

U.S.P. 2,410,605. Interpolymers of Isobutylene With an Alkyl Acetylene and Their Preparation. D. W. Huebner and J. E. Fearey to I.C.I., Ltd.

An interpolymer of isobutylene and methyl acetylene is formed by treating their mixture with a Friedel-Crafts catalyst, such as $AlCl_3$ or BF_3, at a temperature below —10° C.

HYDROGENATION, DEHYDROGENATION, AROMATIZATION

U.S.P. 2,408,146. Dehydrogenation of Hydrocarbons. K. K. Kearby to Jasco, Inc.

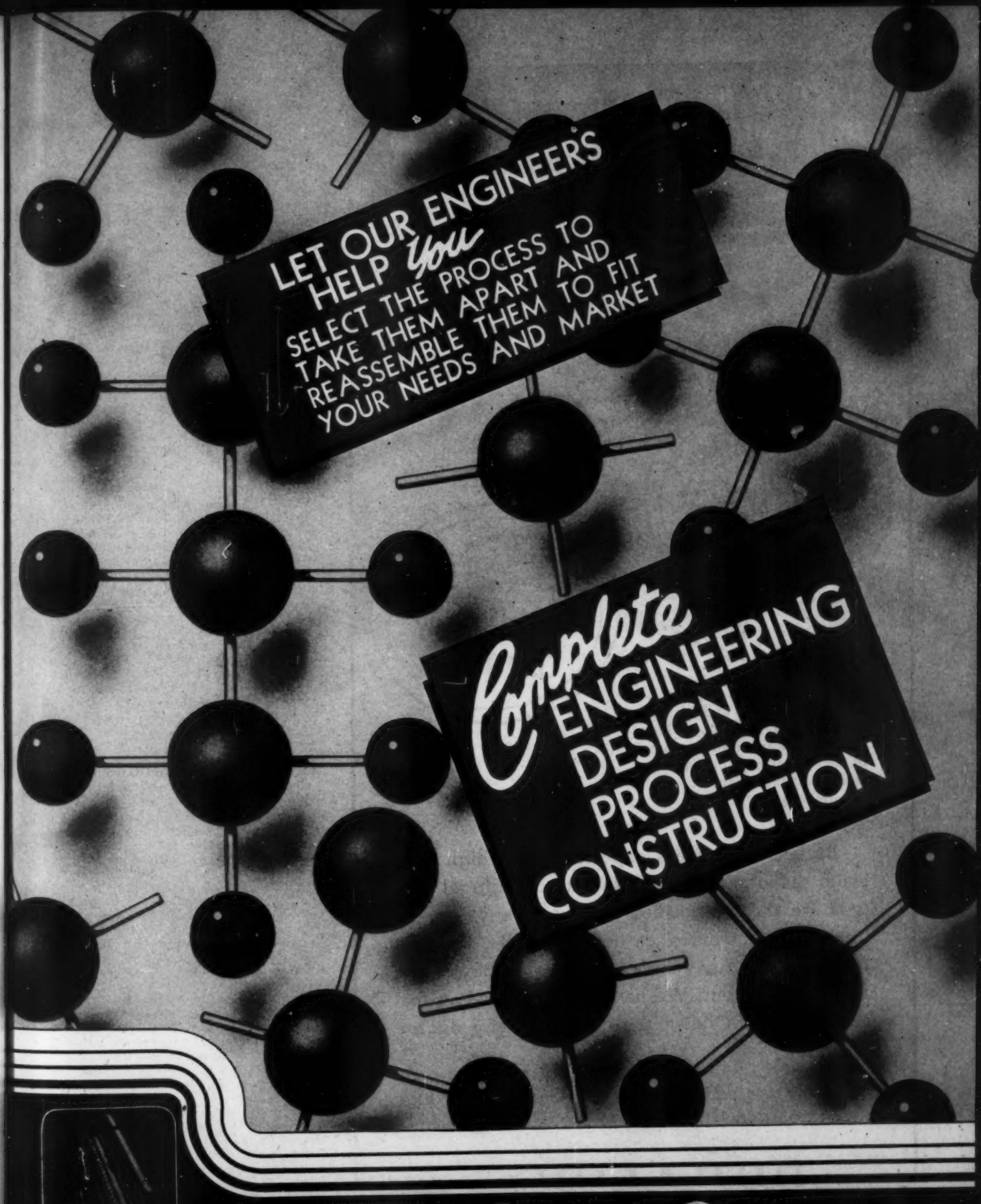
Olefins to be dehydrogenated are diluted with steam and passed at a temperature of 1000-1600° F. over a catalyst which comprises a major proportion of MgO and minor proportions of Cr_2O_3 and KO. The ratio of steam to olefins may vary from 15:1 to 1:1. A suitable catalyst is for example composed of 78 percent MgO, 20 percent Cr_2O_3, 1.5 percent K_2O and 0.5 percent cerium oxide.

U.S.P. 2,408,987. Catalytic Process. M. P. Matuszak and G. H. Morey to Phillips Petroleum Company.

Hydrocarbons are contacted at a temperature below 600° C. with a granular catalyst prepared by subjecting a crystalline ammonium-containing salt of chromic acid, which comprises at least one metal other than Cr, to controlled heating at a temperature below that at which the salt decomposes with incandescence, until the salt is completely decomposed while retaining its crystallic shape. The process which is particularly useful in the dehydrogenation of paraffins changes the C:H ratio of the treated hydrocarbons. The crystalline compound employed in the production of the unglowed metal chromate catalyst may have the formula $(NH_4)_2M(CrO_4)_2.2NH_3$, where M can represent Cu or Cd, or both. The catalyst is superior in catalytic and mechanical properties to catalysts obtained by ignition of chromic acid salts.

U.S.P. 2,409,235. Continuous Process for Effecting Catalytic Reactions. H. V. Atwell to The Texas Company.

A stream of finely divided solid synthesis catalyst containing a carbide-forming metal flows in succession through a primary and then a secondary



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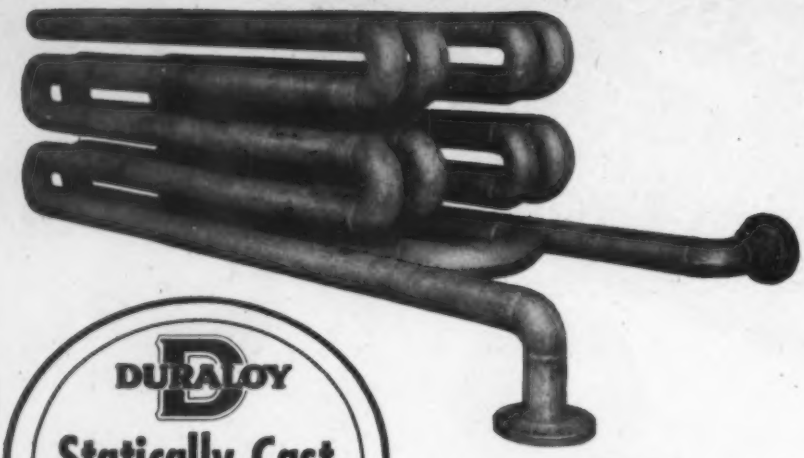

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conversion zone in a continuously recurring cycle. While in the primary zone the catalyst is contacted with CO at an elevated temperature under such conditions that metallic carbide is formed on the catalyst in substantial amount. The carbide-containing catalyst is then contacted in the secondary zone with hydrogen under conditions such that metallic carbide is decomposed and carbon-containing compounds of higher mol weight than methane are formed, particularly liquid hydrocarbons. Co, Fe, and Ni used in the form of a powder are the preferred catalyst metals. A temperature of 350-425° F. is advantageously employed in the first zone and a temperature of 200-400°F. in the second zone.

U.S.P. 2,409,259. Preparation of Conjugated Dienes. T. F. Doumani and R. Deery (now bearing the name R. F. Deering) to Union Oil Company.

A hydrocarbon mixture comprising butanes and butenes is contacted with a catalyst at a temperature of 430°-540° C. and under superatmospheric partial pressure in the absence of added hydrogen. Butadiene and its dimer are produced concurrently. The dimer is depolymerized by subjecting it to a temperature of 600-1000° C. at subasmospheric pressures for a contact time of 0.01-5 seconds, whereafter the reaction is quenched by cooling the reaction products to a temperature below 300° C. within about 0.5 second. The same products and/or other dienes, such as isoprene or cyclopentadiene, and their respective dimers can also be obtained by cracking petroleum hydrocarbon fractions at temperatures of 650-1100° C, and partial pressures of at least one atmosphere, separating from the reaction mixture a fraction boiling below 65° C. from a fraction of higher boiling range and subjecting this latter fraction to the depolymerizing treatment described above.

U.S.P. 2,409,382. Aviation Gasoline Production. E. B. Peck to Standard Catalytic Company.

A cracked hydrocarbon stock containing olefins, naphthenes, and parafins and boiling substantially within the gasoline range is vaporized. The vapors are fractionated to form a light fraction, an intermediate hexane to octane fraction, and a heavy fraction, said light fraction and said heavy fraction containing compounds of relatively high antiknock value as compared with said intermediate fraction. The light fraction and the heavy fraction are blended to gasoline. The intermediate fraction is passed through a heating coil and thence to a hydrogenation zone in which naphthenes are converted into aromatic compounds, and olefins are saturated. A product consisting principally of aromatic compounds and paraffins of 6-8 C atoms is formed. The aromatic compounds are separated from the paraffins and the latter are passed through a heating coil and to a zone where they are converted into a product composed mainly of aromatics. The aromatics produced in the two conversion steps are combined.

U.S.P. 2,409,390. Process for Simultaneously Isomerizing and Separating Cycloparaffins from Acyclic Paraffins by Means of an Aluminum Halide-Hydrocarbon Complex. W. E. Ross and P. Pezzaglia to Shell Development Company.

Cycloparaffins of hydroaromatic structure are produced from a hydrocarbon

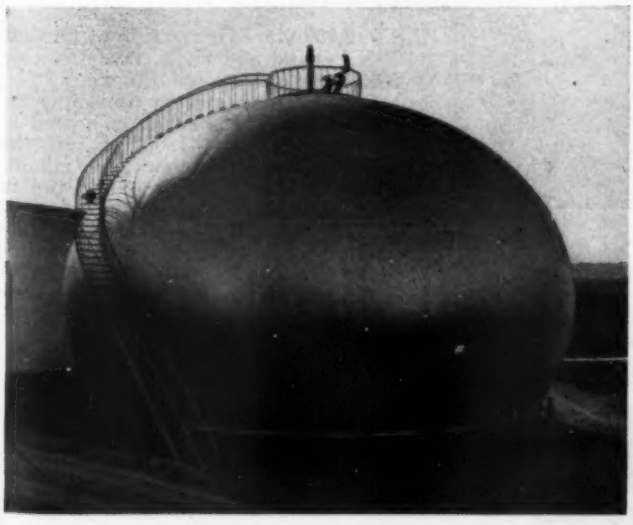
. . . A RUBBER BALLOON HELPED DESIGN . . .

• This is a rubber balloon partly filled with water and subjected to a low internal pressure . . . When placed on a table it takes the form of a spheroid.

The HORTONSPHEROID

. . . the most economical and efficient pressure storage tank for hydrocarbons ranging in volatility from motor gasoline to natural gasoline

• This is a 10,000-bbl. smooth Hortonspheroid . . . Note the similarity in shape with that of the balloon in the photo above.



Take an ordinary toy balloon, partly fill it with water and pump some air into it. Place it on a table and it will take the shape shown in the photo above. The liquid tends to flatten the balloon while the pressure tends to inflate it. The "shell" stresses are uniform.

We used this principle in designing the Hortonspheroid. All of the plates in the shell of the Hortonspheroid, including those in the roof and bottom, are stressed in tension and are thus effective in resisting pressure.

In an ordinary storage tank, the plates in the roof and bottom are not stressed and the cylindrical shell must be of sufficient thickness to resist the entire bursting force of the liquid. This effective use of materials helps to make the Hortonspheroid one of the most economical types of pressure storage tank available.

Smooth Hortonspheroids have been built in capacities from 2,500 to 40,000 bbls. and for gauge pressures as high as 75 lbs. per sq. in. Noded type Hortonspheroids can be built in capacities up to 120,000 bbls. Write our nearest office for Bulletin E describing the Hortonspheroid in detail.

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New York 6 . . . . . 3302—165 Broadway Building	Washington 4 . . . . . 703 Atlantic Building
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HELICOID GAGES
Remain Accurate Longer

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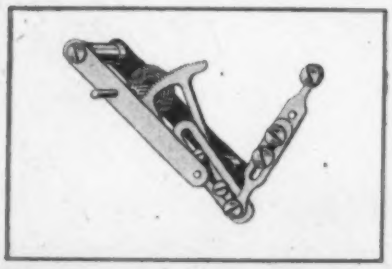
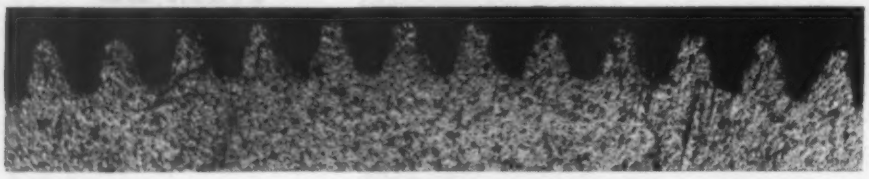
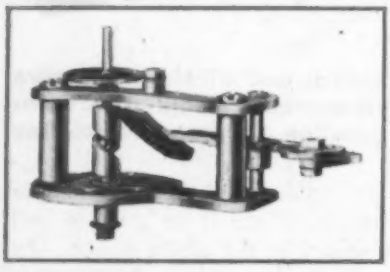


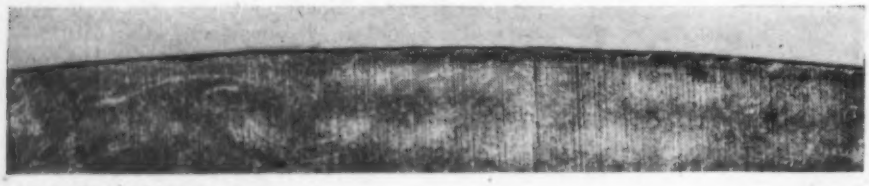
1 The movement of the ordinary pressure gage operates by means of a toothed segment and a spur gear as shown at right.



2 Magnified 12 times, the teeth of this new, unused sector are jagged – anything but uniform. Pinion teeth are cut the same way. Friction, wear and consequent inaccuracies are caused by these rough surfaces rubbing together.



3 Helicoid Gage movement has a polished, graphited Bakelite cam and a polished helicoid roller – instead of sector and pinion.



4 Cam of Helicoid movement magnified 12 times, shows smooth, polished surface. Helicoid roller surface is also highly polished. Longer wear and smoother operation result from these two polished surfaces in rolling, point contact.

Only Helicoid Gages have the Helicoid movement. That is one of several reasons why Helicoid is a better, longer-lasting pressure gage. Write for our complete catalog.

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mixture containing cycloparaffins of non-hydroaromatic structure and open-chain paraffins of about the same boiling range by fractionally distilling the hydrocarbon mixture in the presence of a preformed aluminum halide-hydrocarbon complex and hydrogen halide. A vapor fraction comprising paraffins, and a liquid fraction comprising aluminum halide-hydrocarbon complex and cycloparaffins of hydroaromatic structure is formed.

U.S.P. 2,409,587. Combination Hydrocarbon Dehydrogenation and Hydrogenation Process. A. S. Ramage to Albert A. F. Maxwell.

Hydrocarbons to be converted to other hydrocarbons poorer in hydrogen are contacted in the vapor state in the absence of free reactive oxygen, with a mixture of molybdenum oxide, chromium oxide, and ferric oxide activated by vanadium pentoxide. The temperature of the oxide mixture and vapor is controlled between 800° and 1100° F. so as to oxidize only a part of the hydrogen component of the hydrocarbon. The reaction products comprise principally aromatics and lesser quantities of other unsaturated hydrocarbons, and steam. Part of the olefins may then be hydrogenated to produce lower-boiling saturated hydrocarbons.

U.S.P. 2,409,695. Method for Improving Aviation Fuels. K. C. Laughlin to Standard Oil Development Company.

A naphtha fraction containing naphthenes and added hydrogen is contacted with a molybdenum oxide-alumina catalyst at 850-1100° F. and at elevated pressure. The reaction products are fractionated to recover a fraction rich in aromatics which is subjected to solvent extraction. Aromatics are recovered from the extract phase. The raffinate phase is contacted at 850-1050° F. with a promoted chromia-alumina catalyst and a further quantity of aromatics is recovered from the products of the second reaction.

U.S.P. 2,409,815. Manufacture of Olefins. C. K. Viland and H. Y. Hyde to Tide Water Associated Oil Company.

A hydrocarbon fraction consisting essentially of normal hexane is heated to temperature of 1000-1300° F. in the presence of added amounts of ethane, ethylene, propane, or propylene and in the absence of other added hydrocarbons. The resulting products are fractionated and a mixture of C_6 hydrocarbons is separated. The molal ratio of n-hexane to C_2 and C_3 hydrocarbons subjected to the reaction shall be between about 1:1 and 1:7.

U.S.P. 2,410,044. Hydrocarbon Catalyzing Process. R. E. Burk and E. C. Hughes to The Standard Oil Company (Ohio).

A naphtha containing non-benzenoid hydrocarbons is aromatized by subjecting it in admixture with hydrogen and a hydrocarbon of 4 C atoms at elevated pressure and a temperature of 800-1200° F. to the action of a metal oxide contact-mass formed by co-precipitation from a solution of salts of tin or antimony, aluminum and chromium, or of tin, beryllium and chromium. The tin or antimony, constitute 1-30 mol percent of the contact mass.

U.S.P. 2,410,048. Chemical Process. D. L. Campbell to Standard Oil Development Company.

In the production of butadiene and its

Corkboard insulation holds these chambers within 1 degree of 50° F.

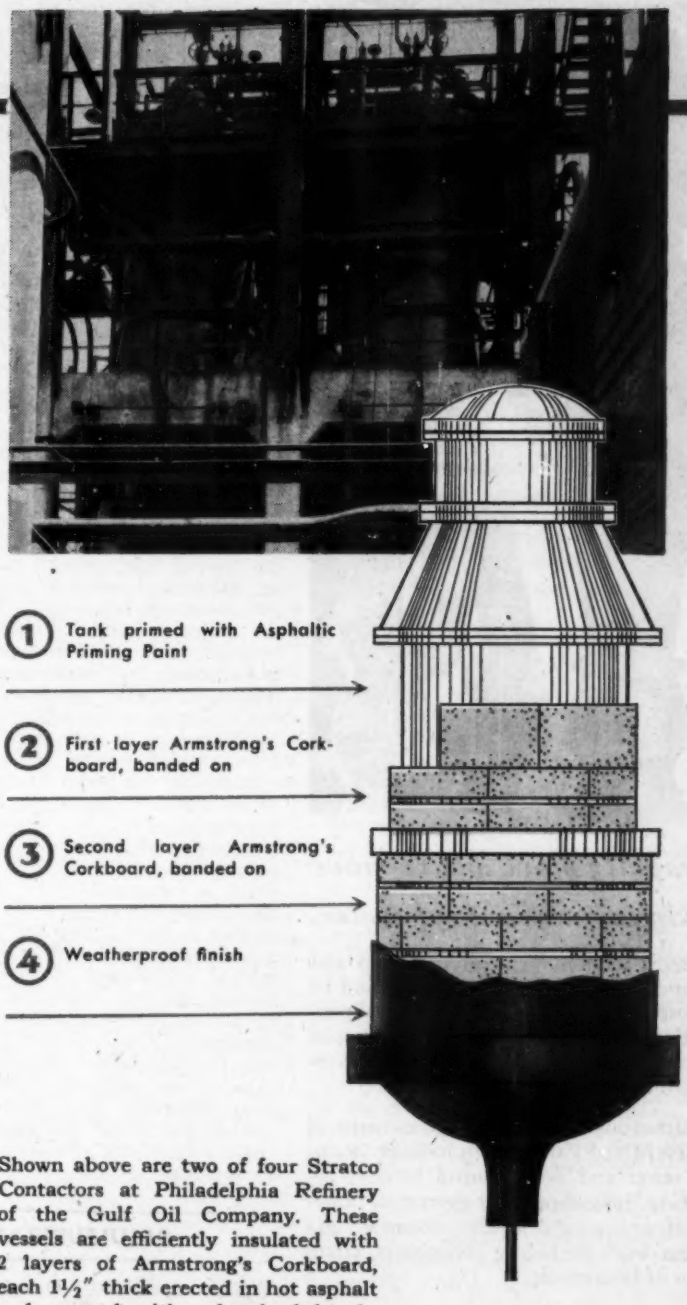
EXTREMELY accurate temperature control can be maintained in low-temperature processing chambers, indoors or out, when they're insulated with Armstrong's Corkboard. Applied in correct thickness, this material virtually eliminates the influence of outside temperatures.

Corkboard and cork covering are particularly efficient insulations in refineries. They have low thermal conductivity and high resistance to moisture. They are light in weight and fire-resistant. Successive layers bond together strongly. Cork can be given weatherproof finishes that have a high degree of fire resistance.

Cylindrical tanks, towers, and other vessels over 54″ o.d. are efficiently insulated with Armstrong's Corkboard scored and bent to fit the contour of the vessel. Corkboard can readily be cut and fitted tightly over irregular shapes.

On cylindrical vessels under 54″ o.d. and over 20″ o.d., Armstrong's Corkboard Lagging and Discs are employed. And for piping and pipe fittings of all sizes from ¼″ up, Armstrong's Cork Covering and Fitting Covers are recommended.

When maintenance of any temperature, low or high, adds to the efficiency of a process, it usually pays to insulate the equipment in which the process takes place. If you have a temperature-maintenance problem, talk it over with one of Armstrong's engineers. Just write to Armstrong Cork Company, Building Materials Division, 7501 Concord St., Lancaster, Pennsylvania.


1 Tank primed with Asphaltic Priming Paint
2 First layer Armstrong's Corkboard, banded on
3 Second layer Armstrong's Corkboard, banded on
4 Weatherproof finish

Shown above are two of four Stratco Contactors at Philadelphia Refinery of the Gulf Oil Company. These vessels are efficiently insulated with 2 layers of Armstrong's Corkboard, each 1½″ thick erected in hot asphalt and secured with galvanized bands. 50° F. is held ± 1° in these chambers.

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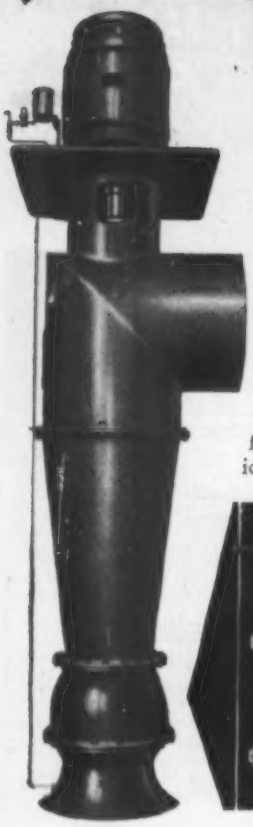
Complete Contract Service For All Temperatures

From 300° Below Zero

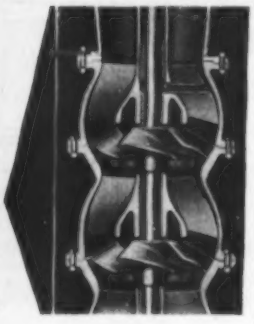
To 2600° Fahrenheit

PEERLESS
Hydro-Foil PUMPS
Move huge volumes of water against low and medium heads



PEERLESS MIXED-FLOW PUMPS
CAPACITIES: UP TO 220,000 G.P.M.

Cutaway view of Mixed-Flow multi-stage pump impeller and bowl construction revealing high efficiency impellers utilized for more economical water lift.



To rapidly move and re-move maximum amounts of water.

The most modern principles of aero and hydro-dynamic design are embodied in Peerless Mixed-Flow pump design and construction, attaining new high efficiencies in large volume water lift and lowering overall maintenance and pumping costs.

Numerous successful applications of Peerless Mixed-Flow Pumps include: drainage; sewer and flood control service; dewatering; irrigation water diversion; flooding of agricultural lands; industrial and process work including condensers, circulation of liquors, etc.

PROPELLER TYPE PUMPS

Peerless pumps, embodying the Hydro-Foil principle, are also available in the propeller type and are capable of handling identical capacities of the Peerless mixed-flow pump. Request Bulletin B-148 on design, construction and application data of these two outstanding types of Peerless Pumps, for large-volume water pumping.

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homologues at relatively high temperatures, for example by dehydrogenation of butene, the hot reaction products from the diolefin formation step are rapidly quenched by contacting them first with water and then with a relatively cool liquid comprising a fat oil. Cooled product gas is separated from the quenching liquid, and a diolefin-containing fraction is liquefied. Uncondensed gases are separated and washed with an absorber oil to recover diolefins therefrom. The fat oil formed in this washing step is utilized in the quenching step.

U.S.P. 2,410,445. Production of Diolefinic Hydrocarbons by Reaction of an Alcohol with an Acetylenic Hydrocarbon. V. N. Ipatieff and H. Pines to Universal Oil Products Company.

A diolefin, such as 1,3-butadiene, is produced by reacting an alkanol, such as ethanol, and an alkyne, such as acetylene, at a temperature of 200-600° C. in the presence of a metal-oxide catalyst having both dehydrating and dehydrogenating activities and consisting of at least one of the oxides of Al, Th, Mg, or Si. A pressure of less than 20 atmospheres is preferably employed in the reaction.

U.S.P. 2,410,642. Process for Oxidation. A. Farkas and A. F. Stribley to Union Oil Company of California.

A saturated cyclic hydrocarbon is treated in the liquid phase with a gas containing free oxygen at temperatures of 200-350° F. and at pressures of 50-300 psi until between 0.1-10 percent of the molecules are oxidized. This proportion of oxidized molecules is thereafter maintained by withdrawing partially oxidized liquid, which is passed to a fractionating column wherein unoxidized hydrocarbon is vaporized and distilled. Unvaporized partial oxidation products containing one atom of oxygen per mol are withdrawn from the bottom of the fractionating column. The vaporized unoxidized hydrocarbon is condensed and returned together with additional quantities of saturated cyclic hydrocarbon to the oxidation step. Exhaust gases and vapors from this step are passed through a condenser and into a separator from which condensed hydrocarbon is returned to the oxidation zone. Partial oxidation products containing the same number of C atoms per mol as the saturated cyclic feed hydrocarbon and containing one oxygen atom per mol are thus obtained. For example, 1-methylcyclohexanol and 2-heptanone or a mixture thereof are obtained from methylcyclohexane.

DESULFURIZATION

U.S.P. 2,409,690. Desulphurization of Hydrocarbon Oils. E. W. S. Nicholson and A. K. Redcay to Standard Oil Development Company.

A mixture of vaporized lower-boiling hydrocarbon oils and hydrogen is continuously contacted with a desulfurizing catalyst at an elevated temperature for a period of time sufficient to materially reduce the amount of sulfur compound in the oil but insufficient to form substantial C deposits on the catalyst. The reaction is preferably carried out under a pressure ranging from atmospheric to 200 psi and at a temperature of 400-800° F. Catalytic material is continuously removed from the treating zone while it contains relatively little sulfur, and is treated in a reactivating zone with hydrogen in an amount and at a temperature sufficient to remove the sulfur from the catalyst, which is then returned to the treating zone.

HEAVY OILS AND WAXES

U.S.P. 2,409,296. Lubricants. R. F. McCleary to The Texas Company.

The lubricant described comprises a hydrocarbon oil and a quantity of less than 5 percent by weight of an oil-soluble sulfurized derivative of partially hydrogenated cardanol, such as a sulfurized derivative of a partially hydrogenated phenolic distillate obtained from the distillation of cashew nut-shell oil. This lubricant possesses enhanced anti-oxidant and anti-corrosive properties.

U.S.P. 2,409,303. Lubricating Oil. J. R. Morris and R. F. McCleary to The Texas Company.

The lubricant according to this patent comprises a mineral lubricating oil having incorporated therein 0.1-5 percent by weight of an oil-soluble metal derivative, particularly a metal salt, of an alkyl substituted aryloxy sulfide and 0.5-5 percent of a sulfurized terpene hydrocarbon. An oil-soluble alkaline-earth-metal salt of an alkyl phenol sulfide and a sulfurized terpinolene can, for example, be used as the additive. The lubricant produced has detergent and anti-oxidant or anti-corrosive properties.

U.S.P. 2,409,333. Lubricant and Method or Preparing Same. D. L. Wright and C. W. Bohmer to Standard Oil Development Company.

The lubricant described in this patent consists of approximately 50-85 percent mineral white oil, 50-10 percent light petrolatum, 0-10 percent paraffin wax, and a viscosity-increasing linear polymer of an oxygen-containing aliphatic compound in an amount selected according to the average mol weight of the polymer. 10-15 percent of a polymerized acrylic acid ester may, for instance, be employed. The lubricant is chemically and particularly nitration-resistant.

U.S.P. 2,409,686.
U.S.P. 2,409,687. Compounded Lubricating Oil. J. G. McNab and D. T. Rogers to Standard Oil Development Company.

The mineral lubricating oil according to this patent contains a small quantity, sufficient to stabilize the oil, of a reaction product of a sulfide of phosphorus with a compound of the formula $(RArX)_2M$, where Ar is an aromatic nucleus, R is an alkyl radical with at least 5 C atoms, X is oxygen, or sulfur, and M is Ca, Sr, Ba, Mg, or Zn. Such a reaction product can, for example, be obtained by reacting elemental S and elemental P with the reaction product obtained from Ba $(OH)_2$ with a tert. octyl phenol.

U.S.P. 2,409,687 for a "Sulfur and Metal Containing Compound" claims the product used, for example, according to U.S.P. 2,409,686 for the stabilization of lubricating oil. The product is useful also for other purposes.

U.S.P. 2,409,726. Lubricant Composition. C. Winning and J. G. McNab to Standard Oil Development Company.

The patent relates to a lubricant suitable for use at high temperatures which comprises a major proportion of a mineral-oil base stock, a minor proportion,

sufficient to impart detergent properties to the lubricant, of a metal salt of a polybasic inorganic acid which contains at least one oil-solubilizing organic group, and a minor proportion, sufficient to inhibit varnish formation, of a higher alcohol. The said metal salt can, for example, be a barium salt of petroleum sulfonic acid, and the alcohol shall preferably contain at least 8 C atoms, as for instance stearyl alcohol.

U.S.P. 2,409,799. Lubricant. E. N. Roberts to Standard Oil Company (Indiana).

The lubricating oil according to this patent contains 0.001-5 percent of a polyamino poly-arylmethane and 0.001-10 percent of a neutralized phosphorus- and sulfur-containing reaction product of phosphorus sulfide and a hydrocarbon, such as an olefin with at least 20 C atoms, e.g., a propylene polymer or an isobutylene polymer.

U.S.P. 2,409,877.
U.S.P. 2,409,878. Lubricating Oil. R. L. May to Sinclair Refining Company.

The petroleum lubricating oil described in this patent contains a minor proportion, e.g., 0.1-2.5 percent by weight of the oil base, of an oxidation inhibitor resulting from the reaction of an alkylated phenol with the condensation product of turpentine and P_2S_5. The alkyl group of the alkylated phenol is a saturated aliphatic redical preferably containing at least 5 C atoms. Diamyl phenol may, for example, be used. The production of the inhibiting compounds is claimed and described in U.S.P. 2,379,312.

According to U.S.P. 2,409,878 the reaction product of zinc oxide with the organic compound used as inhibitor in the preceding patent is employed as oxidation retarding admixture for petroleum lubricating oil.

U.S.P. 2,410,381. Lubricating Oil Composition. V. N. Jenkins to Union Oil Company of California.

The invention relates to a lubricating oil composition which comprises about 25-99 percent by weight of a mineral lubricating oil and about 1-75 percent of a blending agent. This blending agent has a viscosity above 10,000 seconds Saybolt Universal at 210° F. and is produced by chlorinating paraffin wax and dechlorinating the product obtained at temperatures between 400-600° F. without substantial polymerization. Wax olefins thus obtained are distilled to remove any polymerized material formed. The wax olefins are then polymerized and subsequently extracted with a normally gaseous hydrocarbon with 3 or 4 C atoms having high solvent power for paraffin wax and wax olefins and low solvent power for the polymerized products at the temperature of extraction. The polymerized products form the blending agent for the lubricating oil. Anhydrous aluminum chloride may be employed as the catalyst in the polymerization and propane as the solvent for the wax at 170° F.

U.S.P. 2,410,483. Process of Dewaxing Oils. E. M. Dons and O. G. Mauro to Mid-Continent Petroleum Corporation.

A heated stream of wax-bearing oil is introduced into an elongated passageway where it is forcibly mixed with streams of relatively cool dewaxing solvent at successive stages. A cooled solution containing minute wax nuclei crystals at approximately the cloud point of the solution is thus gradually formed. Streams of colder dewaxing solvent are then injected into the steam of waxy solution at successive additional stages. Additional wax is progressively deposited around the minute nuclei formed before, and the dimensions of the individual crystals are gradually increased. The volumes of the streams of colder solvent are progressively increased at consecutive stages to bring about a more rapid growth of the crystals. The linear velocities of the moving mixed stream in these consecutive stages shall be between ¼-3 feet per second. The solution formed is separated from wax crystals by filtration after cooling.

U.S.P. 2,410,578. Mineral Oil Composition. J. J. Giammaria to Socony-Vacuum Oil Company.

The mineral oil composition according to this patent comprises a viscous mineral oil fraction having in admixture therewith a minor proportion of a lignin ester of an aliphatic acid of at least 16 C atoms, such as lignin stearate with at least 3 hydroxyl groups of the lignin esterified with the stearic acid. The quantity of the lignin ester used shall be sufficient to depress the pour point of the oil fraction, 0.05-10 percent of the ester being normally used.

U.S.P. 2,410,613. Flushing Oil. R. F. Ruthruff.

The flushing oil for cleaning the crankcases of internal-combustion engines comprises a fraction from the products of the catalytic reforming of heavy naphtha, which fraction boils within the approximate range of 400-535° F. and exhibits a kauri-butanol value in excess of 100. A mineral lubricating oil of a viscosity within the range 30-300 Saybolt Universal seconds at 100° F. can be admixed.

U.S.P. 2,410,650. Lubricant Composition. J. J. Giammaria to Socony-Vacuum Oil Company.

The lubricant composition comprises a major proportion of a viscous hydrocarbon oil and an oil-miscible metal salt, e.g., a zinc salt of a wax-ayrl-substituted thio acid of phosphorus, such as a wax-substituted diphenyl dithiophosphoric acid, in an amount sufficient to exert detergent and anti-corrosive properties in the oil under heavy duty service. The wax group contains at least 20 C atoms.

U.S.P. 2,410,652. Compounded Lubricating Oil. J. R. Griffin, Jr., and P. R. Van Ess to Shell Development Company.

The low-ash-content lubricating oil for high-temperature internal-combustion engines contains an oil-miscible metal salt of the condensation product of an aldehyde and an aromatic hydroxy compound, such as the calcium salt of the condensation product of formaldehyde or acetaldehyde with alkyl naphthol or octylphenol. This additive is used in an amount sufficient to give high-temperature detergent action to the lubricant, but not in excess to 0.25 percent, based on sulfate ash. An oxidation-inhibiting quantity of an oil-soluble, relatively stable aromatic amine antioxidant is also added to the lubricant, such as 0.1-0.5 percent of naphthylamine or dinaphthylamine.

Saved Costly Delay

A boiler feed pump in a Philadelphia plant ceased to deliver. Inspection showed that the valve seat had become loose, worked up and down, and worn out the threading.

To obtain a new oversize seat would have meant long delay. The engineer remembered Smooth-On No. 1 Iron Cement. He had the worn valve seat cleaned, then reset it with Smooth-On and let it stand overnight.

Next morning the pump went back into regular service. Three years later the valve was reported still working perfectly under 125 lbs. pressure at 200° F.

Smooth-On No. 1 has been sealing cracks, stopping leaks, tightening loose parts and fixtures in mills, plants and factories for the past 50 years. A standby with engineers, mechanics and maintenance men for both emergency and routine repairs. Effective, durable, inexpensive. Your supply house has it in 1-, 5-, 25 and 100-lb. sizes. If you do not find it there, write us.

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1—Pipe-Thickness Gauge

General Electric Company, Special Products Division, Schenectady, New York, has announced a gauge designed to measure the thickness of iron or steel pipes from the outside. Called a pipe-thickness gauge, Type MS-A, the instrument can be used on pipes up to ¼-inch thickness and from 1 to 2 inches in diameter. Applications include checking the safety factor of pipes and tubing carrying petroleum liquids, chemicals, steam, or gas.

The gauge consists of an indicating instrument, a gauge head, and a saddle which is connected to the gauge head by two screws. A different saddle is required for each diameter of pipe. To operate, the saddle is placed upon the pipe, and thickness is indicated in fractions of an inch on the indicating instrument.

The gauge measures the average thickness of a pipe over an area of several square inches. Accuracy of plus or minus 1/64-inch can be obtained on clean surfaces, and 1/32-inch on dirty surfaces. Dirt, scale, and paint on the inside of a pipe do not add to the thickness reading. The gauge head and saddle combination weighs about 6½ pounds. The instrument operates on 115-volt, 60-cycle power.

CHECK THE CARD AT PAGE 200

G-E Pipe-Thickness Gauge, Type MS-A

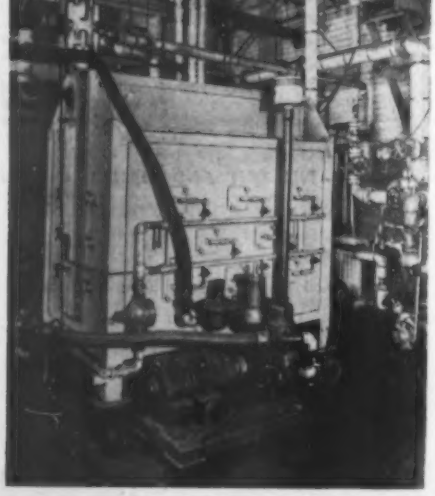
2—Superheaters

Selas Corporation of America, Philadelphia 34, Pennsylvania, manufactures a small and compact superheater with which steam, air and other gases are superheated by a method based on continuous heating principles. Temperatures to 2000° F. are uniformly maintained at pressures limited only by characteristics of heating coils. Design lends itself to adaptation for individual heating requirements. Ideal operating conditions can be approached for each installation. Burners which transmit a high percentage of heat by radiation are patterned on each side of the superheater to produce the desired heat gradient.

A manufacturer of jet apparatus reports favorably on a Selas installation for superheating steam to test jet apparatus at actual operating temperatures and pressures. Saturated steam is fed to the superheater at test pressure and temperature is raised to the value for which the jet is designed. The superheater also is used to heat air for test purposes. Thus, sufficient quantities of steam or air, economically superheated, are available on short notice.

For increased convenience the superheater is mounted on wheels so that it can be moved about the test-bay as required.

CHECK THE CARD AT PAGE 200



Steam is superheated by continuous heating methods in the Selas Superheater. Combustion controller, in foreground, delivers correctly proportioned air-gas mixture at correct pressure to burners.

3—Fans for Corrosive Fumes

The Duriron Company, Dayton 1, Ohio, has issued its Bulletin 1102 on "Durco" corrosion-resistant exhaust fans. It gives engineering data on five standard sizes of fans made by the company, and describes special alloys from which fans can be constructed for special services. Some typical applications are shown by drawings, and information is given on types of drives, as well as hood outlets, bell-end adapters and drip traps.

CHECK THE CARD AT PAGE 200

4—Safety Valve

Oilfield Machine & Supply Company, Box 2172, Houston 1, Texas, is offering a safety valve which is actuated by the velocity of fluids through pipe on its cone-shaped valve stem. It is designed to stop flow in event of a break in the pipe.

The valve is set by one-screw adjustment to pass fluid at a determined velocity. If pressure drop across the valve increases, as in the case of a line break downstream, the increased flow past the cone valve stem pulls the stem into the seat, stopping flow. The valve can be adjusted to close if pressure drop exceeds the desired value as little as three pounds, or as high as required. Reopening is manual. It has only one part that moves, and can be installed in any position. It is available in a variety of sizes, steel or stainless steel.

CHECK THE CARD AT PAGE 200

5—Fire Extinguisher

Walter Kidde & Company, Belleville, New Jersey, has announced production of a new model fire extinguisher containing 5 pounds of carbon dioxide in a recently developed light-weight cylinder. The new portable extinguisher replaces the company's 4-pound type, giving an extra pound of carbon dioxide while knocking off 2½ pounds of the average charged weight—the new model weighs 15½ pounds, while the old 4-pound extinguisher weighed 18 pounds.

Increased carbon dioxide content in the new model, it is pointed out, provides added protection to small hazard

Kidde Light-Weight Extinguisher

areas. Easier handling has been made possible by the reduction in weight. The company has available other portable models in 2½-, 10-, 15- and 20-pound sizes, and wheeled units in 50-, 75- and 100-pound sizes.

CHECK THE CARD AT PAGE 200

6—Needle Valve

Kerotest Manufacturing Company, Pittsburgh 22, Pennsylvania, is making a new precision needle valve for gauge and metering lines, instrument lines and other high-temperature, high-pressure or corrosive service.

Union nut construction prevents blowouts during opening or repacking and also provides an extra deep stuffing box. Positive back seating allows the valve to be repacked under full line pressure. A metal-to-metal line seal at the bonnet eliminates gaskets. Full port clearance prevents interference with flow characteristics by the nipples.

The new valve combines positive shut-off with micrometer throttling action. The ball seat, which provides a positive line seal for complete shut-off, has a tapered tip, actuated by fine stem threads, which permits control of flow and also ease of operation at all pressures.

The valve stem, including ball seat and tapered tip is constructed of specially heat-treated and work-hardened stainless steel, ground and polished—to eliminate possible galling and wire drawing. The valve body is machined from solid rolled stock. The handwheel is an oblong iron casting which provides a firm, easy grip.

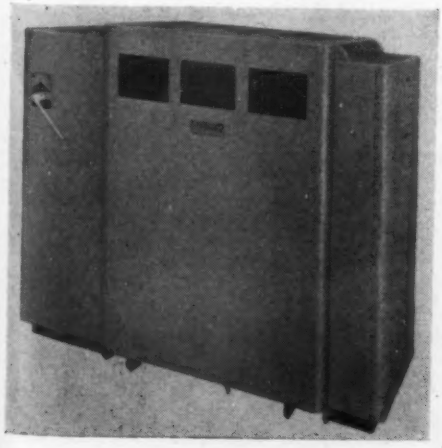
These valves are available in both globe and angle types in sizes from ⅛- to ¾-inch for working pressures up to 3000 pounds; temperatures up to 750° F. They are made from cadmium plated carbon steel, 12-14 percent chrome—Type 416 or 18-8 stainless steel—Type 316—or special alloys to met specifications.

A bulletin containing complete details may be obtained by writing.

CHECK THE CARD AT PAGE 200

7—Unit Substations

Wagner Electric Corporation, 6400 Plymouth Avenue, St. Louis 14, Missouri, has announced its "All-in-One" unit substations, in which high-voltage incoming line, transformer, and low-voltage feeder sections are combined in one compact assembly. It is completely enclosed, streamlined construction, with air-cooled transformer for indoor installation. "Noflamol" (noninflammable-liquid filled) or oil-filled self-cooled transformers also are available.



Wagner "All-in-One" Unit Substation

"All-in-One" unit substations are standardized in ratings 100, 150, 200, and 300 kva, 3-phase, 60 cycles. Choice of combinations of different types of transformers and high- and low-voltage equipment makes them suitable for a wide range of applications. Complete information is available by writing.

CHECK THE CARD AT PAGE 200

8—Demineralizer

Cochrane Corporation, 17th Street and Allegheny Avenue, Philadelphia 32, has announced a line of four package-unit demineralizers for providing industrially pure water. The units are designated by reaction-tank diameters as CDM-12 (12-inch-diameter reaction tank), CDM-18, CDM-24 and CDM-36. The units are designed for plug-in operation from any 110-volt 60-cycle source, and are attractively finished. Units are constructed so that operation and observation is from a position in front of the panel; chemical tanks also are charged from this position, making the unit suitable for location against a wall or in a corner. Control valves and instruments are on the panel at eye elevel. Various reaction materials are applicable to particular conditions and those best fitting raw water and effluent requirements are used.

The manufacturer's Publication 4266 gives full details.

CHECK THE CARD AT PAGE 200

9—Pneumatic Transmission System

Taylor Instrument Companies, Rochester, New York, has given the name "Transet" to a new pneumatic transmission system it announces as particularly adapted to applications involving long lead lines between transmitter and control panel, and where compactness of control panel is a consideration. It is applicable to the control of pressure, rate of flow, and liquid level.

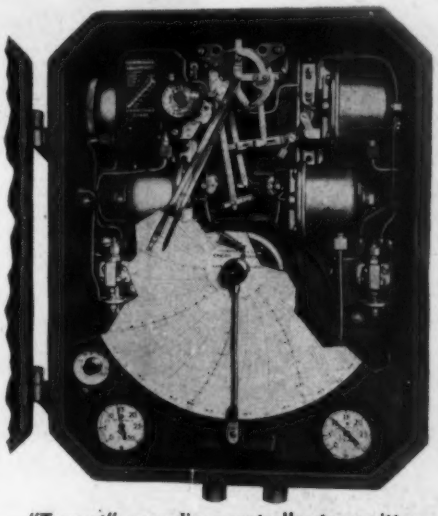
The controller regulates the control valve and also transmits air pressure, proportional to the pen movement, to the indicating receiver remotely located on a panel board. The left-hand gauge of the receiver unit is calibrated in the same units of measurement as the controller chart, thus providing an indication of the controlled variable. The right-hand gauge of the receiver gives an indication of the set pointer position. Thus when both pointers of the receiver unit coincide, the pen and set pointer of the Transet Controller will be together and the process will be under control.

The controller set pointer is pneumatically adjusted by manipulating the reducing valve on the right end of the receiver panel until pointer of pneumatic set gauge is at the desired value.

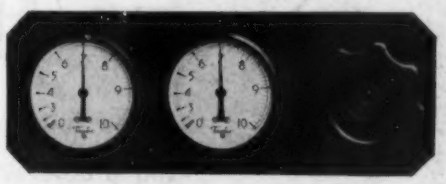
Precise control is achieved by eliminating, or reducing to a minimum, the time lag in the control circuit, because the controller may be located adjacent to the point of measurement.

The receiver is so compact that seven of them take up no more space than two conventional recording receiver-controllers. This means greater economy, especially on large installations, and greater convenience for the process engineer who can quickly check all related process variables without having to move all over a large control room.

Extremely compact, the standard Taylor case contains not only the "Transet" controller, transmitter and pneumatic set



"Transet" recording controller-transmitter



Indicating receiver (left), set pointer (center), set point adjuster (right).

mechanisms, but also allows ample space for either a spring-driven or explosion-proof electric chart clock.

CHECK THE CARD AT PAGE 200

10—Pirani Tubes

Sylvania Electric Products, Inc., Electronics Division, 500 Fifth Avenue, New York 18, has announced availability of Pirani tubes suitable for pressure gauges and leak detectors in evacuating apparatus and automatic pressure recording equipment.

The tubes are designed with special tungsten filaments having a high temperature coefficient of resistance permit-

ting direct readings of gas pressures. Change in gas pressure produces a change in thermal conductivity, filament temperature and filament resistance. Measurement of filament resistance, calibrated in terms of pressures for individual gases, is indicated on an O-1 milliameter placed in a simple bridge circuit.

Matched pairs of Pirani tubes are recommended for greater accuracy. One tube is sealed directly into the evacuating system while the second or compensating tube is left intact, opened to surrounding air or filled with a specific gas to a standard pressure. Tubes usually are mounted in close proximity to provide the same ambient and the installation is usually carefully shielded from radiant heat or air currents.

Readings may be obtained with ± 5 per cent accuracy within a pressure range of 1 to 1000 microns. The tubes are supplied in nonex glass envelopes with tubulation for direct sealing into apparatus. Overall length of the tube is approximately 8 inches. They are designed to operate at 1.5 volts, 100 milliamperes. Cold resistance of the filament is 6.6 ohms. Hot resistance at 100 milliamperes in evacuated tube ranges from 15.5 to 17.0 ohms.

CHECK THE CARD AT PAGE 200

11—Tube Cleaner Head

A recent development by Airetool Manufacturing Company, 300 South Center Street, Springfield, Ohio, permitting easy replacement of floating pins and center plates in expansion-type tube-cleaner heads has been drawing favorable comment from users, the company says.

Airetool P-Type Single Expansion Cutter Heads

The center plate, which controls the extent of expansion in the cutter head, and the pins can be replaced on the job without special tools. The floating pin also serves to reduce the heat generated in cutters by high speeds of the cleaning motor.

The head, designated as the P-Type Single Expansion, is made with combination cone and two or more cylinder cutters in each group. Each group is composed of three or more cutters depending on cutting surface and size required.

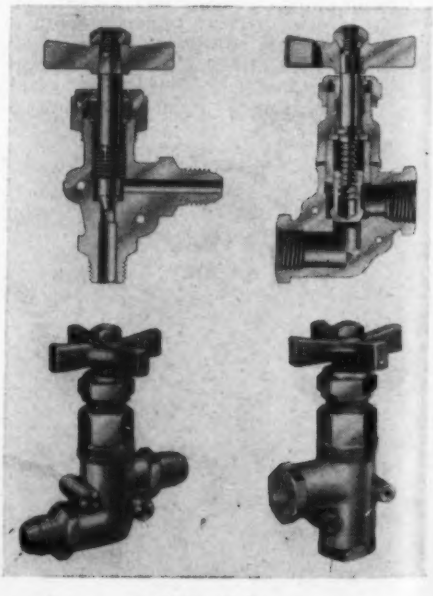
CHECK THE CARD AT PAGE 200

12—Valves

Parker Appliance Company, 17325 Euclid Avenue, Cleveland, Ohio, has announced production of a series of small valves claimed by the maker to incorporate a number of design features new to this field.

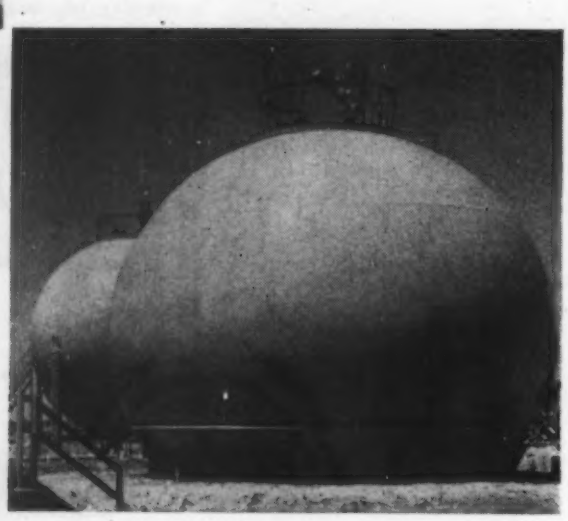
Available in needle and globe types in sizes up to 1-inch, and in both angle and offset designs, the new Parker valves are unusually small and compact by reason of forged-brass bodies. The forging process produces a strong grain structure which makes possible a lightweight, well proportioned design with high strength.

Both the needle and shut-off types are available in six variations of connection



Parker Valves

LITTLE JOB



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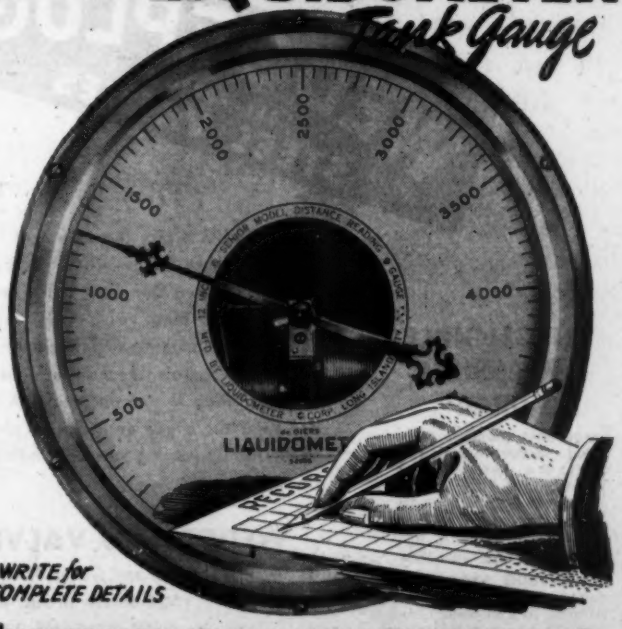

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arrangements. Straight-line types are produced with female pipe threads, or with tubing threads for the use of flexible tubing. Angle types are provided with female pipe threads, with tube threads, or with male pipe threads at the inlet connection and either tube or female pipe threads at the side outlet. These variations enable the new valves to be directly connected into either pipe or flexible-tube systems without adapters. Tubing is accommodated in a range of from ¼- to 1-inch outside diameter, pipe from ⅛- to 1-inch.

All valves in the series are provided with integral mounting lugs for convenience in securing valves to brackets or panels to insure long-life, leak-proof connections in installations where vibration is a factor. Bolt holes are drilled to accommodate standard-size bolts.

An important reduction in pressure drop has been achieved in the offset valve design, by the elimination of pockets, angle ports and intricate chambers. The streamlined path provides free-flow characteristics which under test have proven to decrease pressure drop in the valve by 50 percent over conventional designs, it was said.

The shut-off valves are of the non-rising stem type, and can be repacked under full-flow or no-flow conditions.

Both types of valves are furnished with Duronze stems, standard dimension packings, and easy-to-grasp star handles.

CHECK THE CARD AT PAGE 200

13—Steel Lubricated Plug Valves

American Car and Foundry Company, 30 Church Street, New York 8, is distributing its ACF Valve Catalog No. S-46, giving engineering details on its line of steel lubricated plug valves.

The All-Purpose Valve Has Large Port Openings

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14—Protective Clothing

The B. F. Goodrich Company, Akron, Ohio, has published a new bulletin on its line of industrial protective clothing. It tells of new products introduced in recent months, pictures each garment, describes its functions, and gives data on materials and construction.

CHECK THE CARD AT PAGE 200

15—Hose Masks

Mine Safety Appliances Company, Braddock and Meade Streets, Pittsburgh 8, has issued an illustrated Bulletin EB-5, giving information on its combination hose masks with double inhalation tubes. Blowers and accessories also are described and illustrated.

CHECK THE CARD AT PAGE 200

16—Control Valves

The Foxboro Company, Foxboro, Massachusetts, has issued Bulletin 277-1, "Pneumatic Control Valves and Controller Accessories." Included is a color page identifying finishes offered on valves, corresponding with the color code of the American Standards Association; plates and tables of specifications for control valves, needle type valves, poppet valves and butterfly valves; sections on the Vernier Valvactor for high-accuracy positioning of valve plungers, and on air switches and sub-panels for remote valve control. Information is given on computing valve sizes, with tables and formulas for determining size for a contemplated installation. Air-filter sets, venting dampers, and other accessories are described with illustrations.

CHECK THE CARD AT PAGE 200

17—Hose

Hewitt Rubber, 240 Kensington Avenue, Buffalo 5, New York, has available a descriptive bulletin covering its line of industrial hose, including tank-truck, tank-car, propane-butane, gasoline pump, and oil suction and discharge hose.

CHECK THE CARD AT PAGE 200

18—Automatic Chemical Feed

Milton Roy Company, 1300 E. Mermaid Avenue, Chestnut Hill, Philadelphia 18, is distributing a reprint from transactions of the American Institute of Chemical Engineers: "Electronics in Automatic Chemical-Feed Systems," by Robert T. Sheen, consulting engineer for the company.

CHECK THE CARD AT PAGE 200

19—Rotary Displacement Meters

Roots-Connersville Blower Corporation, 900 West Mount Street, Connersville, Indiana, has released Bulletin 40-B-14, on positive displacement meters. This is a 16-page booklet in two colors, illustrated with shop and installation views, sectional drawing, and proof curves of meter tests. Construction features and advantages are discussed at some length, and a table of sizes, capacities, etc., is included. Roots-Connersville Meters are built for pressures of 25, 50 and 125 pounds, in the standard sizes, and steel-encased meters, for line pressures of 300 pounds upward, are also available on special order. Thirty-nine meter sizes are listed in the table, ranging from 2½ x 7 to 32 x 96 inches. Applications in which these meters have been used include station send-out and city-gate service, industrial and large consumer service, coke-oven and blast-furnace gas, handling butane and propane, and for the measurement and proportioning of many fluids, such as air, hy-

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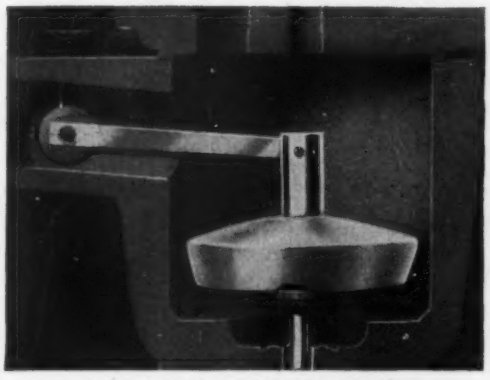

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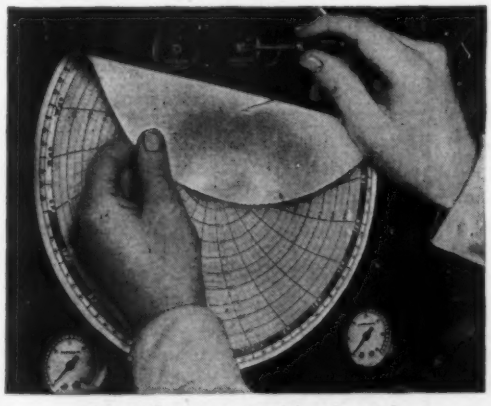

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drogen, oxygen, acetylene, methane, etc. Special applications are discussed, such as laboratory testing, and others. Meter accessories, such as the P. V. T. T. recorder, maximum-demand recorder, pressure and temperature integrator, indicating and recording tachometer, and other instruments, are described and illustrated, along with an explanation of their uses.

CHECK THE CARD AT PAGE 200

20—Packing

Greene, Tweed & Company, North Wales, Pennsylvania, has issued its Catalog No. PC-100, which contains information on service applications as well as construction and performance features of the company's line of packings.

CHECK THE CARD AT PAGE 200

21—Water Conditioning

Allis-Chalmers Manufacturing Company, Milwaukee 1, has issued a new 12-page instruction booklet, entitled "Allis-Chalmers Water Conditioning Chemicals and Equipment," written for the guidance of power-plant operators.

CHECK THE CARD AT PAGE 200

22—Oil Filters

The Briggs Filtration Company, Bethesda 14, Maryland, has available a bulletin descriptive of its line of oil filters for fuels, lubricants, hydraulic oils and coolants. Construction of filters, filter elements and performance for a number of applications are discussed.

CHECK THE CARD AT PAGE 200

23—Gaskets

Flexitallic Gasket Company, 8th and Bailey Streets, Camden, New Jersey, is distributing a booklet which discusses "How Flexitallic Precision Brings a New Degree of Safety for Sealing Steam and Other Liquids Under Pressure."

CHECK THE CARD AT PAGE 200

24—Check Valves

The Williams Gauge Company, 1620 Pennsylvania Avenue, Pittsburgh 12, is distributing a bulletin descriptive of the Williams-Hager Flanged Silent Check Valve. Dimential details in tabular form are included, together with a cutaway view showing construction of the valve.

CHECK THE CARD AT PAGE 200

25—Water Conditioning

Liquid Conditioning Corporation, 423 West 126th Street, New York 27, is distributing Bulletin G, 60 pages descriptive of methods and apparatus for conditioning water and other liquids. It describes all types of water-conditioning processes and explains the applications, advantages and limitations of each. Included are tables listing gaseous and solid impurities, showing the effects, limits of tolerance for various purposes, methods of removal and residual amount of each impurity after treatment, and a comparison chart showing the chemical results produced by various water-treatment methods. Illustrations show diagrammatically the principles of construction and operation of the various processes. Other tables and charts relating to water treatment add further to the usefulness of the bulletin.

CHECK THE CARD AT PAGE 200

26—Welding and Cutting

Air Reduction Sales Company, 60 East 42nd Street, New York 17, has published a new 64-page general welding and cutting products catalog. It includes oxyacetylene welding and cutting gases, equipment and supplies, and arc-welding machines, accessories and electrodes.

27—Acid- and Alkali-Resistant Pumps

Worthington Pump and Machinery Corporation, Harrison, New Jersey, has issued a new bulletin, W-350-B1H, of Worthington Type CG acid- and alkali-resistant pumps. Additional sizes have been added to this manufacturer's line, increasing the coverage to 200 feet and capacities to 1200 gallons per minute. All sizes are production items in Worthite, a stainless steel resistant to a wide range of corrosive conditions. The 20-page illustrated bulletin covers specifications, applications, sectional diagrams, performance curves, etc.

CHECK THE CARD AT PAGE 200

28—Protective Coating

United Chromium, Inc., 51 East 42nd Street, New York 17, has a four-page descriptive folder on "Ucilon," a material developed from inert synthetic resins, and described as providing protective coating for severest corrosive conditions. Properties, application data and typical uses are given.

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About EQUIPMENT—SERVICE Suppliers

George P. Hough Heads Chicago Office of A. O. Smith Corporation

A. O. Smith Corporation has announced that George P. Hough has been appointed executive administrator of the company's Chicago district, taking the place of Don T. Allen who resigned recently.

G. P. Hough

Hough has been with the company since 1939. He entered the Chicago office in that year to direct the sale of pressure vessels.

His activities kept pace with the increasing sales volume of the Chicago office, as the company expanded into such fields as welding electrodes and equipment, and consumer products such as glass-lined water heaters and domestic coal stokers. In 1944 he was appointed assistant administrator in direct charge of several sales activities. Today, with six product sales managers and a force of 35, he will direct all sales for the company's products in the Middlewestern states.

Hough was graduated from the law school of the University of Minnesota in 1920 and was admitted to the bar in Minnesota. He did not practice law, however, but went into sales work with Montgomery Ward & Company. Later he entered the portable phonograph business, in which he was engaged in Milwaukee until 1932. He then went with the Charles E. Hires Company, first in Milwaukee and then in Chicago. After that he became a sales executive for the Northwest Engineering Company of Green Bay, Wisconsin.

Girdler Licensing Agent for Continuous Grease Processes

The Votator Division of The Girdler Corporation recently announced its appointment as licensing agent for the continuous grease processes of Standard Oil Development Company.

Hitchcock Heads Quaker Oats Company Chemicals Department

Lauren B. Hitchcock has been appointed vice president in charge of the chemicals department of The Quaker Oats Company. Since 1944 Dr. Hitchcock has served as manager of the chemicals department in the development of furan chemicals. Formerly he was with Hooker Electrochemical Company as manager of sales development.

L. B. Hitchcock

Frank F. McKinney recently was made sales manager of the chemicals department of The Quaker Oats Company, to head technical sales of furan chemicals. After two and a half years service with the Army, McKinney returned in March of last year to his position with the chemicals department.

Fredus N. Peters, Jr., former director of research, has been appointed vice president in charge of chemical research. Dr. Peters has been in charge of nutritional and organic chemical research for many years and has made major contributions in the field of furan chemistry.

Peavy to Supervise Dresser Products Sales in Southwest

Elton E. Peavy recently has been appointed Southwest district manager for the sale of Dresser products. This assignment includes responsibility for the sale of couplings, fittings and repair products in Texas, Louisiana, Oklahoma, Arkansas, Mississippi and New Mexico.

E. E. Peavy

Peavy succeeds H. R. Shidel who will direct sale of Dresser welding fittings in the area and supervise the Houston warehouse which serves the Southwest with a complete line of Dresser Manufacturing Division products.

Vapor Recovery Systems Appoints Cleveland Manager

Vapor Recovery Systems Company, Los Angeles, has opened a direct factory sales and engineering office at 1501 Euclid Avenue, Cleveland, Ohio, with T. L. Reibling as district manager. Reibling is a mechanical engineer and will handle the company's business throughout Western New York, Western Pennsylvania, Ohio, Kentucky and Virginia.

Arcos Corporation Opens New Welding Laboratory

Arcos Corporation of Philadelphia has completed a new laboratory building adjoining its plant to house its research and engineering department. The new laboratory facilities will permit the research and engineering department to expand its research and quality control activities.

Drummond and Bennett Elected Mathieson Vice Presidents

The Mathieson Alkali Works recently elected D. W. Drummond its vice president-general manager of sales, and Arthur T. Bennett vice president-manager of operations.

Drummond joined the Mathieson organization in 1938 as assistant sales manager of the carbon dioxide division; a year later he was named sales manager of the division, then assistant general manager of sales of the company, and, at the time of his election to his new post, was general manager of sales. Drummond attended the University of Pennsylvania from which he graduated in 1926.

D. W. Drummond

A. T. Bennett

Bennett obtained his education at Cass Technical School in Detroit, Michigan, Detroit University and Syracuse University. In 1933, he joined Mathieson as a technical assistant. Subsequently, he served as superintendent of the soda ash department at Mathieson's Lake Charles, Louisiana, plant, as assistant manager at the Saltville plant, assistant director of operations in New York, and, in 1943, became general manager of operations.

Clayton Mark & Company Names Carnes to Mid-Continent Post

Joe R. Carnes, Jr., has joined the sales organization of Clayton Mark & Company, Evanston, Illinois, to represent the company in the Mid-Continent oil areas. Carnes will make his headquarters at 1752 West Main Street, Houston 6.

Joe R. Carnes, Jr.

Carnes recently was discharged from the Army Air Forces with the rank of captain, after service in the European theater where he was a bomber pilot and also flew unarmed over enemy territory as a courier pilot. It has taken him five years to realize his ambition of entering the selling profession because the Army got him immediately after he finished school.

Project Engineering Company Moves to Larger Quarters

Project Engineering Company has moved its general headquarters to larger quarters at 115 Fulton Street, New York 7. Offices at 15 Maiden Lane are being retained and mail may be addressed to either location.

The company maintains fabrication shops at Long Island City, New York.

Clark Bros. Company Makes Additions to Official Roster

Clark Bros. Company has announced addition of the following members to the roster of its official family:

F. H. Light, vice president, treasurer and secretary; J. N. MacKendrick, vice president in charge of engineering; D. K. Hutchcraft, vice president in charge of sales, and F. W. Laverty, general sales manager.

Light has been with the company for 35 years, during which he has served consecutively as purchasing agent, treas-

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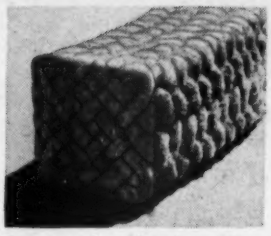

Cross section, showing unique structural design.

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60th Anniversary
1887—1947

GARLOCK
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PACKING

LARGEST FLEXIBLE BELLOWS EXPANSION JOINT

This is a natural photograph reproduced without re-touching, showing a shop view of the largest flexible bellows type expansion joint ever built. It has recently been put into service in one of the nation's largest plants for making aviation gasolene by the Catalytic Cracking process. It is built according to the exclusive FLEXODISC design using corrugated alloy steel plates welded together at their inner and outer edges by the General Electric Atomic Hydrogen welding process. Hundreds of FLEXODISC joints are in service in petroleum refineries and other plants where dependability and bottle-tight construction are exceptionally important.

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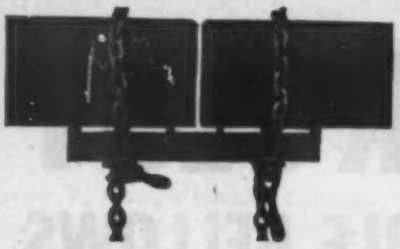

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F. H. Light

F. W. Laverty

D. K. Hutchcraft

J. N. MacKendrick

urer and secretary. Hutchcraft joined the company in 1926, as a salesman, later becoming Mid-Continent sales manager which office he held until 1945 when he became general sales manager. MacKendrick came to Clark Bros. in 1929 as engine designer and served in that capacity for the next seven years when he succeeded C. Paul Clark as chief engineer.

Laverty, entered Clark service in the foundry, in 1927. Thereafter, he served successively as test and development engineer, advertising manager, technical service manager, technical sales manager and assistant general sales manager.

No change was made in the balance of the company's officers.

E. B. Badger & Sons Company Expands Organization

E. B. Badger & Sons Company has announced the following additions to its facilities serving the petroleum, petrochemical and chemical industries:

Re-establishment of E. B. Badger & Sons (Great Britain) Ltd., with offices at 2 Great Winchester Street, London, E. C. 2, England. J. T. T. Robinson is director and general manager.

Establishment of a Brazilian company, Badger Servicos Tecnicos de Engenharia S. A., with offices at Rua Buenos Aires 135, Rio de Janeiro, Brazil. Officers of this company include Dr. Roger H. Newton, director-president; Ernest V. Owen, director-technical, and Dr. Eduardo Guinle Filho, director-financial.

Appointment of Fluor-Lewis and Company, comprised of J. S. Fluor and Milton Lewis, as its Western United States representative, with offices at 416 Petroleum Building, 714 West Olympic Boulevard, Los Angeles 15, California.

Baker Retires After Long Service With McKee & Company

R. E. Baker resigned, effective January 1, as secretary and treasurer of Arthur G. McKee & Company, Cleveland. To succeed him H. R. Moorhouse has been elected as secretary, and James Krumhansl as treasurer. Moorhouse has been Baker's assistant since 1929 and in recent years has been assistant secretary of the company. Krumhansl has been associated with the company since 1913 and for many years has been assistant treasurer.

Baker retires after 40 years of continuous service with the company and its predecessors. He has been a director, secretary and treasurer of the company since its incorporation—first as a Pennsylvania Corporation in 1915, and later, in 1920, as a Delaware corporation. He became a partner of Arthur G. McKee soon after the latter opened an engineering office in Cleveland in 1905, and has been one of the principals in building the business of the McKee Company. He will continue as a member of the advisory committee of the board of directors.

Tracy Elected Vice President Oil Well Supply Company

Lloyd E. Tracy has been elected vice president of Oil Well Supply Company, United States Steel Corporation subsidiary, and will continue as general manager of sales with headquarters at Dallas, Texas.

Lloyd E. Tracy

Tracy has been with Oil Well Supply Company since early in 1935, having transferred to that organization from National Tube Company with which he had been connected since 1927. He was appointed general manager of sales of Oil Well Supply Company in September, 1943.

Texas Electric Steel Castings Names Eastern Representative

J. F. Bechtle has been named Eastern sales representative for Texas Electric Steel Casting Company, Houston, and will maintain offices at 7 East 42nd Street, New York.

Bechtle formerly was associated with The M. W. Kellogg Company from 1923 to 1943, serving with the research and development, engineering and sales divisions. He also served at one time as assistant general manager for Standard Steel Corporation, Los Angeles.

Texas Electric Steel Casting Company recently added complete heat treating facilities for fabrication work to its services for manufacturers and refineries.

Schwanhausser Again Heads Diesel Engine Manufacturers

E. J. Schwanhausser, Worthington Pump & Machinery Corporation, was reelected president of the Diesel Engine Manufacturers Association at the annual meeting in Chicago. J. E. Peterson, General Machinery Corporation; and Gordon Lefebre, Cooper-Bessemer Corporation, were reelected vice presidents. Robert H. Morse, Jr., Fairbanks, Morse & Company, was again elected treasurer. Harvey T. Hill, who has been executive director for the association the past three years, was appointed to continue in that capacity.

Directors elected were William E. Corrigan, American Locomotive Company; George W. Codrington, General

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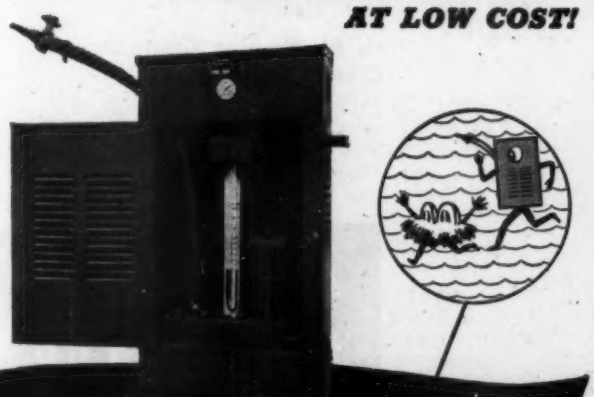

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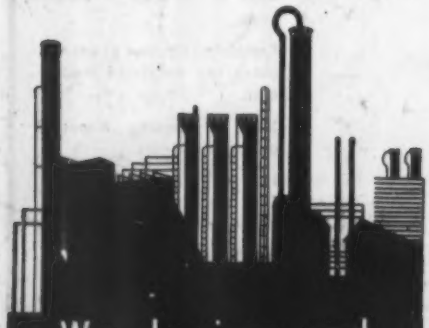

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Carl L. Zak Named Manager of Sales for Pittsburgh Steel

Pittsburgh Steel Company announced the promotion of Carl L. Zak from assistant sales manager to general manager of sales. Zak joined the company in 1945, and before that was for three years secretary of Seamless Steel Tube Institute. He also had six years of sales engineering, inspection and mill scheduling for Globe Steel Company, and another six years with Youngstown Sheet & Tube Company in various sales capacities.

Carl L. Zak

L. A. Ver Bryck has been named assistant manager of sales. He has been district manager of sales in the New York office, and will be succeeded in that capacity by Joseph G. Smith, who has been manager of Pittsburgh district sales. Smith will be replaced by Walter D. Schlundt, former district sales manager at Detroit. Edward L. Dull will become Detroit sales manager. He has been connected with the company's Chicago office. Assistant district sales manager at Pittsburgh will be Robert W. Mullin.

Monsanto Chemical Company Announces Five Promotions

Appointment of Robert B. Semple, of St. Louis, as director of its general development department has been announced by Monsanto Chemical Company in naming five major promotions in company management.

Semple will be succeeded as petroleum-chemicals sales manager by J. W. Newcombe of the petroleum sales department.

William G. Krummrich was named assistant division general manager of the company's Organic Chemicals Division. Krummrich will be succeeded as division production manager by David L. Eynon, Jr., assistant to the division general manager and acting plant manager of the company's Nitro, West Virginia, unit.

Semple attended Principia School in St. Louis and Massachusetts Institute of Technology, where he received a B.S. in chemical engineering in 1932 and a master's degree in chemical engineering the following year. He joined Monsanto as a research chemist in the Organic Division in St. Louis, and in 1934 served as a pilot-plant chemist and from 1935 to 1939 as a pilot-plant group leader. In 1939 he became assistant director of the general development department at St. Louis and was named petroleum chemicals sales manager in 1944.

Newcombe was educated at De Pauw University where he graduated in 1930. He served with the Indiana State Highway Commission and the D-A Lubricant Company both of Indianapolis, before joining Monsanto May 1, 1944.

Krummrich began his career after one year of high school, when he went to work in a stove foundry and studied business courses in night school, and chemical engineering while a member of a fire department. He joined Monsanto in 1917, doing analytical work in the laboratory of the John F. Queeny plant in St. Louis, and rose to operating superintendent of the Queeny plant in 1935. He was transferred the next year to the Monsanto, Illinois, plant as manufacturing superintendent and was made plant manager about four years later. In 1943 he became production manager of the organic division.

Eynon came to Monsanto in 1933 in the research department of the Merrimac Division at Everett, Massachusetts. During the war he was plant manager of the government-owned Longhorn Ordnance Works at Karnack, Texas, which was operated by the company's organic division. Eynon was educated at Williams College and at Massachusetts Institute of Technology.

Graver Tank & Manufacturing Absorbs Banks Moreland

Graver Tank & Manufacturing Company, East Chicago, Indiana, and Banks Moreland Company, Houston, Texas, have announced that the tank business of the latter has been purchased by the former, and will continue to operate in the Southwest as Banks Moreland Company, a division of Graver Tank & Manufacturing Company. The division will be under the management of Banks Moreland who has been made a vice president of Graver Tank & Manufacturing Company.

Brown Instrument Company In New Location at Tulsa

The Brown Instrument Company has opened a new branch office at 117 West Eighth Street, Tulsa, in charge of R. P. Walker as manager. The new location includes storage space for instrument accessories, supplies and emergency repair stock.

H. M. Harper Company Opens Branch in Dallas

H. M. Harper Company, Chicago, recently has moved its branch facilities from Houston to 302 Wilson Building, Dallas, Texas. The new office is under the supervision of W. H. Kreer.

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